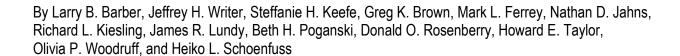


Prepared in cooperation with the Minnesota Pollution Control Agency

Endocrine Disrupting Chemicals in Minnesota Lakes—Water-Quality and Hydrological Data from 2008 and 2010



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U.S. Department of the Interior

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## **Conversion Factors**

#### Inch/Pound to SI

Multiply	Ву	To obtain
	Length	
centimeter (cm)	0.3937	inch (in)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
	Area	
square kilometer (km ² )	0.3861	square mile (mi ² )
	Volume	
liter (L)	0.2642	gallon (gal)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound, avoirdupois (lb)
	Pressure	
torr	0.0193	pound per square inch (PSI)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:  $^{\circ}F=(1.8\times^{\circ}C)+32$ 

Vertical coordinate information is referenced to the North American Vertical Datum of 1988 (NAVD 88). Altitude, as used in this report, refers to distance above the vertical datum.

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius ( $\mu$ S/cm at 25°C). Concentrations of chemical constituents in water are given either in milligrams per liter (mg/L) or micrograms per liter ( $\mu$ g/L).

## **Abbreviations**

AP, alkylphenolethoxylates

SPE, octadecylsilica solid-phase extraction

CASRN, Chemical Abstract Services Registry Number

CLLE, continuous liquid-liquid extraction

EVAP, evaporated to dryness

GC/MS, gas chromatography/mass spectrometry

GC/MS/MS, gas chromatography/tandem mass spectrometry

LAS, linear alkylbenzene sulfonate

MDL, method detection limits

NA, not available

NRPL, National Research Program Laboratory

SIM, selected ion monitoring

™, trademarked

USGS, U.S. Geological Survey

--, not determined

## **Units**

cm, centimeter °C, degree Celsius

hr, hour km, kilometer

km², square kilometer

L, liter m, meter

mg/L, milligram per liter

mg/L C, milligram per liter carbon

mL, milliliter

mL/min/m², milliliters per minute per square meter

mm, millimeter

ng/L, nanogram per liter

nm, nanometer

 $\begin{array}{ll} \text{Std units,} & \text{standard pH units} \\ \mu\text{g/L,} & \text{microgram per liter} \end{array}$ 

μm, micrometer

μS/cm, microsiemens per centimeter

v/v, volume/volume <, less than

# Endocrine Disrupting Chemicals in Minnesota Lakes—Water-Quality and Hydrological Data from 2008 and 2010

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#### **Abstract**

Understanding the sources, fate, and effects of endocrine disrupting chemicals in aquatic ecosystems is important for water-resource management. This study was conducted during 2008 and 2010 to establish a framework for assessing endocrine disrupting chemicals, and involved a statewide survey of their occurrence in 14 Minnesota lakes and a targeted study of different microhabitats on a single lake. The lakes ranged in size from about 0.1 to 100 square kilometers. varied in trophic status from oligotrophic to eutrophic, and spanned a range of land-uses from wetlands and forest to agricultural and urban use. Water and sediment samples were collected from the near-shore littoral environment and analyzed for endocrine disrupting chemicals, including trace elements, acidic organic compounds, neutral organic compounds, and steroidal hormones. In addition, polar organic compound integrative samplers were deployed for 21 days and analyzed for the same organic compounds. One lake was selected for a detailed microhabitat study of multiple near-shore environments. This report compiles the results from the field measurements and laboratory chemical analysis of water, sediment, and polar organic compound integrative sampler samples collected during 2008 and 2010. Most of the organic compounds measured were not detected in any of the water samples, although a few compounds were detected in several of the lakes.

#### Introduction

Most research on the occurrence and effects of endocrine disrupting chemicals (EDCs) in the aquatic environment has focused on effluents from wastewater treatment plants (WWTPs) and effluent-impacted streams (Folmar and others, 1996; Jobling and others, 1998; Tyler and others, 1998; Barber and others, 2007; Vajda and others, 2008; Lee and others, 2010). However, a variety of natural and synthetic EDCs can enter lake environments via sources such as: runoff

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from surrounding urban and agricultural land use, septic system discharges from residences along the shoreline, and recreational activity (Swartz and others, 2006; Conn and others, 2010; Writer and others, 2010; Lee and others, 2011). In general, the most important estrogenic chemicals in the aquatic environment include the natural and synthetic steroidal hormones  $17\beta$ -estradiol, estrone, and  $17\alpha$ -ethynylestradiol, and the alkyphenol compounds 4-nonylphenol and 4-*tert*-octylphenol (Van den Belt and others, 2004; Bistodeau and others, 2006; Sumpter and others, 2006; Barber and others, 2007; Schoenfuss and others, 2008).

Although the occurrence of EDCs in effluents and streams (and associated endocrine disruption in fish) has been documented, there is less information on the occurrence and impacts of EDCs in lakes that do not receive WWTP effluent discharges. For example, Lake Mead (Arizona and Nevada), which receives the discharge from a number of upstream WWTPs, has been shown to have elevated concentrations of synthetic chemicals and endocrine disruption in common carp (Rosen and others, 2006). The potential susceptibility of lake ecosystems to endocrine disruption has been documented by a rapid collapse in fish populations across multiple species after dosing a pristine Canadian lake for several years with approximately 5 nanogram per liter (ng/L) of the synthetic estrogen 17α-ethynylestradiol (Palace and others, 2002; Kidd and others, 2007). However, after the 17α-ethynylestradiol dosing was terminated, the fish populations recovered (Palace and others, 2009). Based partially on data presented in this report, Writer and others (2010) describe the low-level but widespread occurrence of EDCs and fish endocrine disruption in Minnesota lakes that do not have WWTP effluent inputs. Endocrine disruption also has been reported in fish from Lake Thun, Switzerland (Bogdal and others, 2009). Skelly and others (2010) have described endocrine disruption in frogs from urban ponds. These results suggest the widespread occurrence of EDCs and fish endocrine disruption in lakes that do not receive WWTP effluent discharges.

## **Purpose and Scope**

The purpose of this cooperative study between the Minnesota Pollution Control Agency (MPCA) and the U.S. Geological Survey (USGS), which included collaboration with St.Cloud State University (SCSU), was to determine the occurrence of endocrine disrupting chemicals in water and sediment from lakes across the State of Minnesota. This report compiles data from a multi-tiered investigation into the occurrence of EDCs in Minnesota lakes conducted during 2008 and 2010 that consisted of: (1) a statewide survey of the chemistry of 14 lakes (11 sampled during 2008 and three sampled during 2010; two sampled both years), and (2) a detailed microhabitat study to characterize the hydrology and chemistry of the littoral zone in Sullivan Lake. Sullivan Lake is characteristic of many Minnesota lakes influenced by the surrounding landscape that includes agricultural land-use and residences using septic systems for onsite wastewater disposal. Results from the detailed study can be used to better understand the results from the statewide survey.

Companion investigations into endocrine-disruption in wild-caught fish and controlled field-exposure experiments using caged fathead minnows were conducted in conjunction with the water sampling in the 11 lakes sampled in 2008 (Ferrey and others, 2008) and in Sullivan Lake during 2010 (Poganski, 2011; Schoenfuss and Poganski, 2011).

#### **Methods**

#### **Statewide Survey**

During 2008 and 2010, a statewide survey of 14 lakes representing a range of physiographic, geological, and land-use characteristics of Minnesota (fig. 1; table 1) was conducted to evaluate the occurrence of endocrine disrupting chemicals. The lakes covered a range of trophic status (oligotrophic to eutrophic) and anthropogenic inputs from agriculture and urban activities. Several sites were in remote locations with minimal development, including Northern Light Lake, Elk Lake, Beast Lake, Ryan Lake, and Bohall Lake. Between May 22 and August 3, 2008, water was collected during single sampling events from 11 lakes, starting in the southern part of the state and moving northward. Sediment samples from the same lakes were collected approximately one month later. Polar organic compound integrative samplers (POCIS; Alvarez and others, 2004) acquired from Environmental Sampling Technologies (St. Joseph, Missouri) were deployed during the summer of 2008 (at the time of water sampling) for 21 days at 11 of the lakes. During 2010, water samples were collected from three additional lakes and two of the lakes sampled in 2008 (Elk Lake and Sullivan Lake) were resampled.

Grab samples from the water surface were collected using a pre-cleaned stainless steel bucket rinsed with lake water and processed following USGS sampling protocols (U.S. Geological Survey, variously dated). Samples were transported on ice to the USGS National Research Program Laboratory (NRPL) in Boulder, Colorado where they were stored at 4 degrees Celsius (°C) until analysis. Approximately 1 month after the water sampling, composite sediment samples were collected from the top 10 centimeters (cm) directly beneath the bed surface in close proximity to the sites where water and POCIS samples were collected. The sediment samples were stored frozen until analysis. The POCIS samplers were attached at a depth of 1 meter (m) to existing docks or to steel posts located approximately 5 m offshore. Upon retrieval, the POCIS samplers were placed in sealed cans and transported to the NRPL where they were stored at 4°C until analysis. Water samplings and POCIS deployments were conducted at the same locations and during the same time periods as companion biological studies conducted by SCSU on endocrine disruption in wild-caught fish and caged fathead minnows (Ferrey and others, 2008).

### Sullivan Lake Microhabitat Study

To provide a better understanding of the spatial and seasonal relationships between contaminant sources and processes occurring in the littoral zone, Sullivan Lake was selected for the detailed microhabitat study. Sullivan Lake has a relatively small watershed (3 square kilometers (km²)) with underlying glacial morphology that is characteristic of many Minnesota lakes. Sullivan Lake (fig. 2) is located near the town of St. Cloud in central Minnesota and is classified as meso-eutrophic (moderate biological productivity), has a surface area of 30 hectares (73 acres), and has a maximum depth of 16.5 m (Minnesota Department of Natural Resources, 2010). The bottom substrate varies from sand to detritus, but primarily is muck and silt. Predominant land use in the Sullivan Lake watershed was 49 percent cropland, 11 percent deciduous forests, 8 percent developed (residential), 7 percent wetland, and other land-use classifications were 5 percent or less (fig. 2; Minnesota Department of Natural Resources, 2010). The lake may potentially be influenced by residential land use (septic systems), recreational activities such (boating and fishing), and agricultural runoff.

The near-shore environment of Sullivan Lake was evaluated at four microhabitat sites (fig. 2), including two residential influenced areas (sites A and B) with septic systems, a stormwater runoff and recreational activity influenced area (site C), and an agricultural (animal and crop) runoff influenced area (site D) with no residential land use. Site C contained a boat ramp used by lake residents and the general public. Much of the shoreline near site C was covered by thick emergent wetland vegetation, and the bottom sediments consisted of detritus and organic muck. In contrast, lawns extended to the shorelines of the residential/septic sites (A and B) and the near-shore bottom sediments were generally sandy with little or no emergent vegetation. Surface-water samples were collected at each microhabitat site in pre-cleaned and baked amber glass bottles in the same manner as described for the statewide lake survey and shipped to the NRPL, where they were stored at 4°C until analysis. Summer and fall samplings were conducted in order to evaluate seasonal variability.

#### Microhabitat Groundwater/Surface-Water Interactions

The potential for groundwater/surface-water interactions was evaluated at the four microhabitat sites using near-shore transects for pore-water sampling (fig. 2) with ideal (site A) cross-section spacing of 1, 5, 10, and 15 m perpendicular from shore and 1, 3, and 5 m parallel to shore. At site B, dense emergent wetland vegetation prevented sampling at two of the sites closest to shore. Sites C and D had dense emergent wetland vegetation that generally limited sampling the near-shore environment: two locations (1 near the shore and the other 15 m offshore) were sampled at site C, and two transects were sampled at four locations at site D.

Vertical hydraulic-head gradient measurements and pore-water samples were obtained using a hydraulic potentiomanometer (Rosenberry and LaBaugh, 2008) and a retractable well screen inserted to a depth of 30 cm below the sediment/water interface. A peristaltic pump was used to draw water from the surrounding sediments at a rate of less than 10 milliliters (mL) per minute (min) to minimize the hydrodynamic disturbance and avoid excessive degassing or inadvertent additions of lake water to the sample. A manometer was connected to the tubing from the well screen following collection of 60-mL pore-water samples (the pumping period needed to eliminate air bubbles from the system) and measurements of hydraulic-head differences were made at each location. Sediments at sites C were too fine for pulling water through the well screen and precluded measurements of hydraulic gradients. Groundwater flux also was determined at site A using the seepage-meter techniques described in Rosenberry and LaBaugh (2008). Two seepage meters were installed at different points along the shoreline and two measurements were made for each meter.

Field measurements (including determination of temperature and lake depth) were made at the time of each sample collection. Water-column, water/sediment interface, and pore-water temperature were determined using a rigid probe with a thermistor thermometer at the end. Temperature was recorded at the water/sediment interface ( $T_{\text{water/sediment}}$ ) just above the bed surface and 30 cm below the water/sediment interface ( $T_{30 \text{ cm}}$ ). *In situ* water-quality measurements (specific conductance, dissolved oxygen, and pH) were taken with a field probe at various depths in the water column along a 5-point transverse transect parallel to the shoreline (fig. 2) at each microhabitat site.

#### **Chemical Analysis**

Field measurements, including temperature, pH, specific conductance, and dissolved oxygen were made according to standard procedures (U.S. Geological Survey, variously dated).

Dissolved (1-micrometer (µm) glass fiber filter) organic carbon was analyzed by platinum catalyzed persulfate/ultraviolet light oxidation with infrared detection (Barber and others, 2001; Weishaar and others, 2003). Linear alkylbenzene sulfonate (LAS) and alkylphenolethoxylates (AP) were analyzed in unfiltered water samples by enzyme-linked immunosorbent assay (ELISA) using standard methods (http://www.abraxiskits.com). Trace and major elements (table 2) were determined in 0.45-µm filtered water samples by inductively coupled plasma-mass spectrometry (Garbarino and Taylor, 1996; Taylor, 2001) and inductively coupled plasma-atomic emission spectrometry (Garbarino and Taylor, 1979). Because a stainless steel bucket was used in the collection of surface-water grab samples, there is the potential for the introduction of low concentrations of select trace metals and thus potential positive bias.

Unfiltered water samples were analyzed for organic constituents using three different analytical methods described in detail elsewhere (Barber and others, 2011). Acidic organic compounds (table 3) were evaporated to dryness (EVAP) and derivatized with 10 percent (v:v) acetyl chloride:propanol to form the propyl esters (Barber and others, 2000). Neutral organic compounds (table 4) were isolated using continuous liquid-liquid extraction (CLLE) with methylene chloride at pH 2 following ionic strength adjustment (Barber and others, 2000). Steroid and steroidal hormone compounds (table 5) were isolated by octadecyl silica solid-phase extraction (SPE) followed by elution with methanol, Florisil cleanup, and derivitization with N-methyl-N-trimethylsilyltrifluoroacetamide (Barber and others, 2011; Foreman and others, 2012).

The CLLE and EVAP extracts were analyzed by electron impact gas chromatography/mass spectrometry (GC/MS) in both the full scan and selected ion monitoring (SIM) modes. The general chromatographic conditions were: Hewlett Packard (HP) 6890 GC; column - HP Ultra II (5 percent phenylmethyl silicone), 25 m × 0.2 millimeter (mm), 33-µm film thickness; carrier gas, ultra high purity helium with a linear-flow velocity of 27 cm per second (sec); injection port temperature, 300°C; initial oven temperature, 50°C; split vent open, 0.75 min; ramp rate, 6°C/min to 300°C; hold time, 15 min at 300°C. The mass spectrometer conditions were: HP 5973 Mass Selective Detector; tune with perflurotributylamine; ionization energy, -70 eV; source pressure, 1×10⁻⁵ torr; source temperature, 250°C; interface temperature, 280°C; full scan, 40 to 550 atomic mass units (amu) at 1 scan/sec. Concentrations were calculated based on SIM data using diagnostic ions for each compound, which was identified based on matching of retention times (plus or minus 0.05 min) and ion ratios (plus or minus 20 percent) determined from analysis of authentic standards. External calibration curves and internal standard procedures were used for calculating concentrations.

Gas chromatography/tandem mass spectrometry (GC/MS/MS) analysis of the derivatized SPE samples was conducted using a Waters QuattroMicro QqQ GC/MS/MS with an Agilent 6890 gas chromatograph. Chromatography was on a 30 m  $\times$  0.25 mm  $\times$  0.25  $\mu$ m Restek Rtx-XLB capillary column at a He flow rate of 1 mL/min with the injection port maintained at 275°C. The gas chromatograph was programmed on a variable temperature gradient from 100°C to 310°C. For each compound, the most abundant ion in the electron impact spectrum was selected as a precursor, and appropriate conditions were selected to maximize signal for three precursor-product transitions per compound. Quantification was based on external calibration curves and internal standard procedure.

Target compounds in sediment were isolated by accelerated solvent extraction (ASE) with isopropyl alcohol and cleaned up on a Florosil column (Burkhardt and others, 2005). The sediment extracts were analyzed by GC/MS as for CLLE, and then derivitized and analyzed by GC/MS/MS as for SPE. The OASISTM hydrophilic-lipophilic-balance SPE sequestration medium

encased in the POCIS samplers was transferred into a glass column containing silanized glass wool and eluted with methanol. The methanol extracts were evaporated under nitrogen, augmented with dichloromethane:methanol (95:5 v:v), and cleaned up on Florosil column, followed by GC/MS analysis as for CLLE. The extract was then derivitized and analyzed by GC/MS/MS.

#### **Quality Assurance**

The quality-assurance program consisted of field duplicates, field samples spiked with target compounds, field blanks, distilled water spiked with target compounds, and distilled water blanks. Target compounds spiked into the distilled water and natural water matrices were processed in the same manner as environmental samples and used to evaluate method recoveries. Recoveries for individual samples also were evaluated using surrogate standards spiked into each sample prior to isolation. No standard reference materials were available for the organic compounds evaluated in this study. For the EVAP and CLLE GC/MS, surrogate standards (tables 3 and 4) were added to the water samples prior to extraction and analysis and results are presented as percent recoveries. Target compound quantitation for the CLLE and EVAP GC/MS analysis used internal standards and multi-point calibration curves. Standards were obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany), Cambridge Isotope Laboratories (Cambridge, Massachusetts), Sigma-Aldrich (Milwaukee, Wisconsin), and Supelco (Bellefonte, Pennsylvania). Deuterated polynuclear aromatic hydrocarbon internal standards were added to the extracts prior to GC/MS analysis. The SPE GC/MS/MS analysis used 12 deuterated standards (table 5) added following sample collection and isotope dilution quantitation protocols (Foreman and others, 2012). The sediment and POCIS samples used the same surrogate standards and quantitation procedures as the CLLE and SPE methods.

Method detection limits (MDLs) for water samples were defined as the concentration equivalent to three times the mean detection value in method blanks or five times the baseline, whichever was greater. For the sediments, MDLs were determined to be three times the mean detection value in method blanks, corrected for sediment mass extracted. For the POCIS samples, MDLs were set at three times the instrument detection values, corrected for number of POCIS extracted. Spike and recovery studies were not done for the POCIS samplers.

## Water-quality and Hydrological Data

### **Statewide Lake Survey**

Results for the chemical analysis of water, sediment, and POCIS samples collected from 11 lake sites during 2008 are presented in tables 6 to 14. Results for the four lake sites sampled during 2010 are presented in tables 15 to 18.

The field measurement (table 6) results for the 2008 sampling indicate that the chemical characteristics differed broadly among the lakes, as would be expected considering they ranged from oligotrophic to eutrophic. For example, specific conductance ranged from 33 microsiemens per centimeter ( $\mu$ S/cm) in oligotrophic Northern Light Lake to 580  $\mu$ S/cm eutrophic Cedar Lake. Trace element results (table 7) varied. Concentrations of boron, which typically is associated with WWTP effluents (Barber and others, 2011) were relatively constant and ranged from 7  $\mu$ g/L in Kabetogama Lake (oligotrophic) to 33  $\mu$ g/L in Budd Lake (eutrophic).

Organic compounds commonly associated with endocrine disruption (see tables 3 to 5) were detected in the water, sediment, and POCIS samples collected during 2008 (tables 8 to 14). Although only a fraction of the organic compounds measured in the water were detected and concentrations were typically at sub-microgram per liter ( $\mu$ g/L) levels, several compounds (including EDCs) had widespread occurrence. For example, ethylenediaminetetraacetic acid, 4-nonylphenol, and estrone were detected in the water of multiple lakes at concentrations ranging from 0.05 to 0.39, 0.11 to 0.21, and 0.0006 to 0.0015  $\mu$ g/L, respectively. Most of the compounds detected in the water also were detected in the sediments, but at much higher concentrations. Likewise, most compounds detected in the water also were detected in the POCIS samples.

Table 15 summarizes field measurement data for the four lakes sampled during 2010. Ryan Lake and Beast Lake had similar water temperature, specific conductance, dissolved oxygen, and pH values. Bohall Lake had lower water temperature (9.6°C) and higher dissolved oxygen (10.0 milligram per liter, mg/L) values. Elk Lake had the highest water temperature (11.8°C) and specific conductance (294 micro Siemens per centimeter,  $\mu$ S/cm) values, and the lowest dissolved oxygen (8.6 mg/L).

Table 16 summarized the acidic organic compounds detected in the lake water samples collected during 2010. Ethylenediaminetetraacetic acid (EDTA) was detected in Elk Lake (0.53 μg/L), Ryan Lake (0.40 μg/L), and Bohall Lake (0.06 μg/L), but nitrilotriacetic acid (NTA) and 4-nonylphenolethoxycarboxylates (4-NPEC) were not detected in any of the lakes. Surrogate standard recoveries ranged from 54 to 67 percent. Neutral organic compounds are summarized in table 17. Trace levels of 4-methylphenol; 2,6-di-*tert*-butyl-1,4-benzoquinone; and 2,6-di-*tert*-butyl-4-methylphenol were detected in several lakes. Surrogate standard recoveries ranged from less than 10 to 85 percent. Steroid and steroidal hormone compound data from lake samples collected during 2010 are summarized in table 18. Cholesterol, coprostanol, and estrone were detected in the lakes. Surrogate standard recoveries ranged from 37 to 115 percent.

#### **Sullivan Lake Microhabitat Study**

### Geohydrological Environment

Analyses of topographic maps, aerial photographs, and field surveys of Sullivan Lake were used to provide preliminary information on potential groundwater/surface-water interactions (Winter, 1998). The landscape around Sullivan Lake is characterized by Pleistocene glacial deposits and appears to have been part of a larger lake (perhaps connecting to Maple Lake; fig. 2) that during glacial times was dammed up behind the 50-foot high, esker-like ridge along the eastern shoreline (fig. 3). The lake stage may have continued to rise until the lake eventually eroded through the ridge creating the narrow, deeply-incised canal-like outlet near the southeast corner of Sullivan Lake. The presence of wetlands adjacent to the north, northwest, and southwest shorelines indicates that hydraulically driven groundwater discharge likely is shifted to the margins between the uplands and wetlands rather than occurring at the boundary between the wetlands and the open-water lake or could be controlled by lake level in topographically low areas. The predominant geologic feature, from the perspective of groundwater discharge into the lake, is the esker-like ridge along the eastern and southeastern shoreline. Higher groundwater head beneath the ridge creates a hydraulic gradient that increases the potential for groundwater discharge along the western shoreline, which coincides with the areas of maximum residential density and creates the greatest potential for onsite wastewater-disposal systems to discharge into

the lake. Storm water and agricultural runoff along the western shoreline is potentially buffered by the wetlands, which may attenuate chemicals before they enter the lake.

#### Microhabitat Characterization

A preliminary shoreline site investigation was conducted during the microhabitat site-selection process and the results are presented in table 19. Based on these results and hydrological considerations, four sampling sites were selected. Microhabitat A (residential/septic influence) had several hydraulic head values that were higher than the lake surface (positive values, table 20) and that ranged up to 15 mm. The hydraulic-head values were generally low near the shore and greatest at sample location (point 1, 3). The near-shore seepage-meter measurements indicated groundwater influx of -0.94 to 4.3 milliliters per minute per square meter (mL/min/m²). Dissolved organic carbon values in the pore water samples ranged from 2.1 to 7.4 mg/L. Linear alkylbenzene sulfonate values in pore water samples ranged from less than 0.02 to 0.92 mg/L. There was only a single AP detection (0.14 mg/L). T_{30 cm} measurements were generally lower than T_{water/sediment} values. Water-quality measurements were taken within the water column in microhabitat A (table 21). Water temperature, specific conductance, and pH were generally constant with depth with the exception of the 15, 5 point. Dissolved oxygen values were either constant within the water column or decreased with depth.

Microhabitat B (residential/septic influence) had several hydraulic-head values that were slightly lower than the lake surface (negative values, table 22), suggesting the potential for flow from the lake to groundwater at this location. Dissolved organic carbon values in the pore-water samples from microhabitat B (7.6 to 11.4 mg/L) were greater than microhabitat A. The LAS values ranged from less than 0.02 to 0.12 mg/L, and there were sporadic detections of AP (up to 0.11 mg/L). No  $T_{30~\rm cm}$  or  $T_{\rm water/sediment}$  measurements were recorded in microhabitat B. The water-column temperature, specific conductance, dissolved oxygen, and pH were relatively constant at microhabitat B (table 23).

Dense emergent vegetation limited access to sampling locations in microhabitat C (stormwater/boat ramp influence). Dissolved organic carbon values of pore-water samples ranged from 7.3 to 11.2 mg/L (table 24). Low levels of LAS (0.03 mg/L) and AP (0.11 mg/L) were detected in pore-water samples. The T_{30 cm} temperatures (17.7 to 19.4°C) were lower than the T_{water/sediment} temperatures (23.7 to 28.6°C). A thick layer of organic muck at the water/sediment interface prevented hydraulic head measurements. Measurements taken within the water column in microhabitat C indicated that water temperature and specific conductance were relatively constant (table 25). Dissolved oxygen concentrations decreased with depth in the water column and near-bottom values were as much as 43 percent lower than near-surface values. The 3, 5 point was an exception where dissolved oxygen and pH were fairly constant with depth.

Dense emergent vegetation and deep water limited sampling in microhabitat D (agricultural influence). A thick layer of organic muck near the water/sediment interface limited hydraulic head measurements (table 26). Linear alkylbenzene sulfonates were detected at concentrations ranging from 0.02 to 0.11 mg/L, and no AP were detected. Values for T_{30 cm} (16.7 to 18.8°C) were lower than T_{water/sediment} (19.9 to 23.8°C). Measurements taken within the water column at microhabitat D (table 27) indicated that temperature was relatively constant and dissolved oxygen and pH decreased with depth (with the exception of 0, 5 where they remained relatively constant). Specific conductance values did not follow the same trends.

#### **Temporal Sampling**

Acidic organic compounds detected in the summer 2010 Sullivan Lake microhabitat sampling are listed in table 28. Ethylenediaminetetraacetic acid, nitrilotriacetic acid, and 4-nonylphenolmonoethoxycarboxylic acid were detected at all of the microhabitat sites. Surrogate standard recoveries ranged from 66 to 98 percent. Acidic organic compounds detected in the fall 2010 sampling are listed in table 29. Ethylenediaminetetraacetic acid was present in all microhabitats, and microhabitat A (residential/septic influence) had the only detection of nitrilotriacetic acid and 4-nonylphenoldiethoxycarboxylic acid. Surrogate standard recoveries ranged from 46 to 70 percent.

Neutral organic compounds detected in the summer 2010 Sullivan Lake microhabitat sampling are listed in table 30. Several compounds were detected at multiple sites, including bisphenol A; 2,6-di-*tert*-butyl-1,4-benzoquinone; 2,6-di-*tert*-butyl-4-methylphenol; N,N-diethyl-*meta*-toluamide; 4-methylphenol; and 4-nonylphenol. Microhabitat A had the highest levels of N,N-diethyl-*m*-toluamide and 4-nonylphenol, and had the only caffeine detection among the four sites. Surrogate standard recoveries ranged from 29 to 99 percent, and matrix spike recoveries ranged from less than 10 to 163 percent. Neutral organic compounds detected in the fall 2010 sampling are listed in table 31. Sites A and B had detections of bisphenol A; 2,6-di-*tert*-butyl-1,4-benzoquinone; 2,6-di-*tert*-butyl-4-methylphenol; N,N-diethyl-*meta*-toluamide; and 4-nonylphenol. 4-*tert*-Octylphenol and caffeine also were detected at sites A and B. Surrogate recoveries ranged from 19 to 57 percent.

Steroid and steroidal hormone analysis for the summer 2010 Sullivan Lake microhabitat sampling are listed in table 32. Cholesterol, coprostanol, and estrone were detected at all microhabitat sites and 4-androstene-3,17-dione was detected at all sites except microhabitat A. Surrogate standard recoveries ranged from less than 10 to 183 percent, and matrix spike recoveries (excluding cholesterol, which had such high background concentrations it was difficult to calculate recoveries) ranged from 15 to 235 percent. Table 33 lists steroid and steroidal hormone analysis of surface-water samples from each microhabitat site collected in the fall of 2010. Cholesterol and coprostanol were detected at all sites, and a single detection of estrone occurred in microhabitat D. Surrogate standard recoveries ranged from 15 to 121 percent.

#### Conclusions

This report presents the results from field measurements and laboratory analysis for water, sediment, and POCIS samples collected from 14 Minnesota lakes during 2008 and 2010. These data provide a snapshot of the occurrence of a variety of potential endocrine-disrupting compounds in the near shore environment of 14 geographically distributed Minnesota lakes covering a range of land use and trophic status. One of the lakes, Sullivan Lake, was studied in detail to assess potential contaminant sources and evaluate the spatial and seasonal relationships.

## **Acknowledgments**

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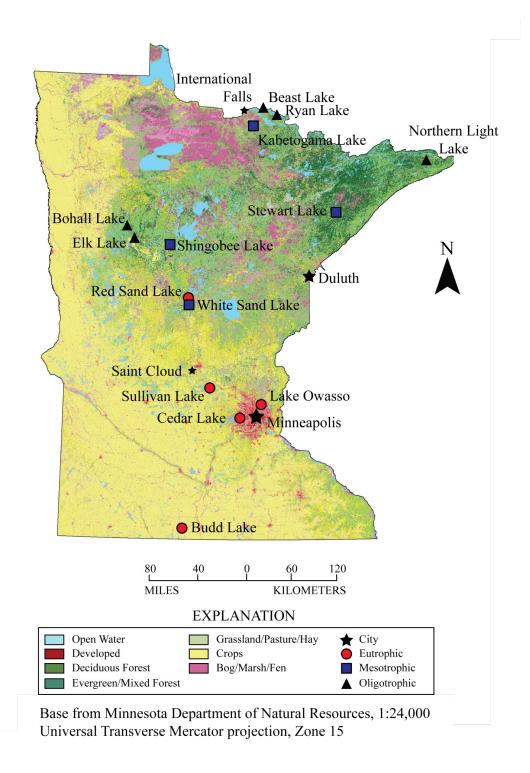
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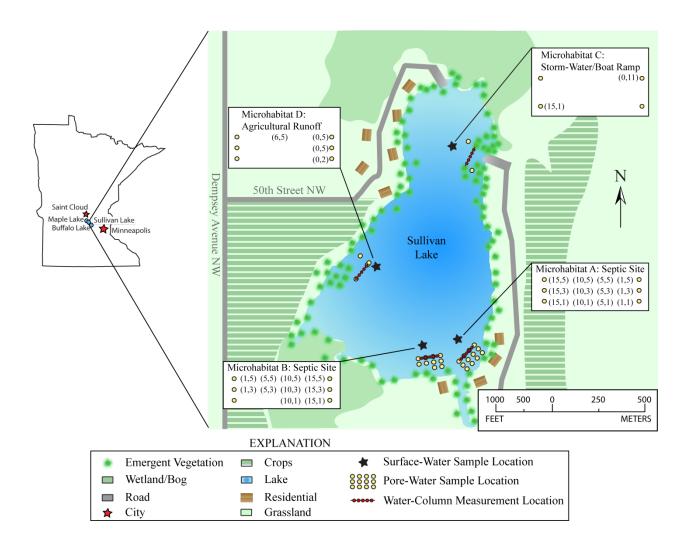
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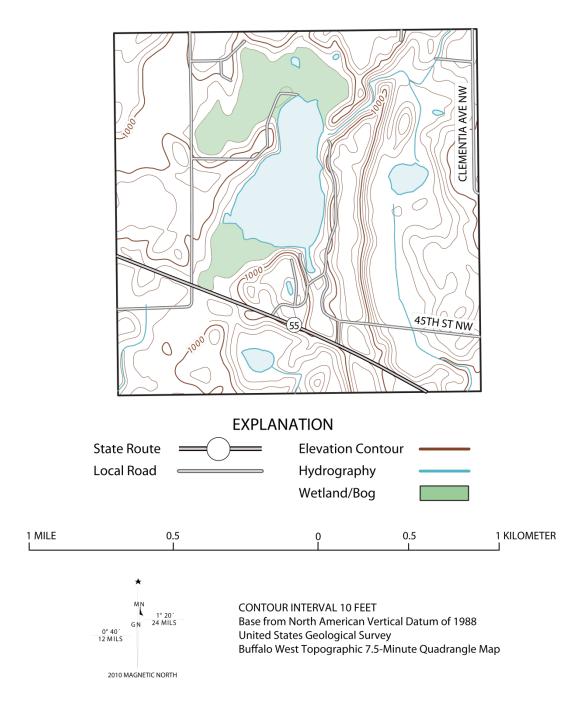
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**Figure 1.** Site locations for the 14 Minnesota lakes sampled during 2008 and 2010 (after Writer and others, 2010).



**Figure 2.** Site locations for the four littoral-zone microhabitats investigated at Sullivan Lake, Minn. Numbers in parentheses (indicate microhabitat groundwater/surface water sampling locations in meters parallel (x) and perpendicular (y) to the shoreline.



**Figure 3.** Site topography and preliminary hydraulic analysis of potential zones of groundwater discharge in Sullivan Lake, Minn.

**Table 1.** Site characteristics for the 14 Minnesota lakes that were sampled in this investigation.

[DOW, Division of Wildlife; km2, square kilometer; m, meter; m3, cubic meter;  $\mu$ g/L, microgram per liter; km, kilometer; n/a, not applicable; Eu; eutrophic; Meso, mesotrophic; Oligo, oligotrophic; POCIS, polar organic compound integrated samplers; --, not available; Date format: M/DD/YY ]

	Unit	0wasso	Cedar	Budd	Red Sand	Sullivan	White Sand	Kabetogama	Shingobee	Stewart	Northern Light	Elk	Beast	Ryan	Bohall
Latitude		45.0313	44.9562	43.6397	46.3810	45.2200	46.3599	48.4442	46.9994	47.1825	47.9081	47.1965	48.5077	48.51881	47.2087
Longitude		-93.1300	-93.3254	-94.4744	-94.2861	-93.9423	-94.2874	-93.0284	-94.6874	-91.7547	-90.2521	-95.2216	-92.7538	-92.7064	-95.2484
Lake DOW number		62005600	27003900	46003000	18038600	86011900	18037900	69084500	29004300	38074400	16008900	15001000	69083700	69083500	15000900
County		Ramsey	Hennepin	Martin	Crow Wing	Wright	Crow Wing	St. Louis	Hubbard	Lake	Cook	Clearwater	St. Louis	St. Louis	Clearwater
Hydrologic class		Headwater	Drainage	Drainage	Drainage	Headwater	Headwater	Riverine	Drainage	Drainage	Riverine	Drainage	n/a	n/a	n/a
Watershed area	$\mathrm{km}^2$	12	10	107	18	3	5	717	42	5	478	35	n/a	n/a	n/a
Lake surface area	$\mathrm{km^2}$	1.64	.72	.94	2.17	.31	1.81	104	.75	1.03	1.97	1.33	0.33	0.14	0.15
Maximum depth	m	11.3	15.5	5.18	7	16.5	7.9	23.8	11	6.1	2.3	28.4	20.1	3.7	NA
Lake volume	$m^3$	5.0E+06	4.0E+06	3.1E+06	2.3E+06	1.3E+06	5.0E+06	7.3E+08	4.0E+06	1.9E+06	2.3E+06	1.3E+07	n/a	n/a	n/a
Volume/ watershed area		.410	.395	.029	.125	.420	.983	1.023	.096	.388	.005	.364	n/a	n/a	n/a
Shoreline	m	9.1E+03	4.9E+03	3.6E+03	6.1E+03	2.7E+03	5.4E+03	1.8E+05	4.9E+03	5.1E+03			n/a	n/a	n/a
Trophic index		Eu	Eu	Eu	Eu	Eu	Meso	Meso	Meso	Meso	Oligo	Oligo	Oligo	Oligo	Oligo
Total phosphorous	$\mu g/L$	39	24	89	26	48	21	15	31	14	13	11	23	12	NA
Septic systems	number	0	0	0	68	42	138	100	3	3	0	0	0	0	0
Docks and marinas	number	164	5	80	32	6	85	242	1	13	0	1	0	0	0
Shoreline septic density	per/km	.0	.0	.0	11.2	15.4	25.6	.6	.6	.6	.0	.0	0	0	0
Shoreline dock density	per/km	18.1	1.0	22.3	5.2	2.2	15.8	1.4	.2	2.6	.0	.2	0	0	0
Total length of streams	km	2,508.9	112.2	48,797.0	1,953.3	15.3	.0	391,614.1	6,907.3	24.2	227,398.2	5,010.6	n/a	n/a	n/a
Total length of roads	km	94,722.2	95,536.9	163,712.4	54,841.7	6,841.4	23,179.0	97,550.2	48,231.9	400.4	92,969.5	6,283.6	n/a	n/a	n/a
Total population	persons	13,227.0	13,329.7	5,805.3	2,162.2	168.9	1,345.3	264.3	391.4	1.5	51.5	2.0	n/a	n/a	n/a
Population density	person/km	1,078.2	1,327.9	54.3	117.4	55.1	264.6	.4	9.3	.3	.1	.1	n/a	n/a	n/a
Road density	km/km ²	7.7	9.5	1.5	3.0	2.2	4.6	.1	1.1	.1	.2	.2	0	0	n/a
Stream density	km/km²	.205	.011	.457	.106	.005	.000	.546	.164	.005	.476	.141	n/a	n/a	n/a
Developed cover	percent	73	80	10	10	8	19	0	4	0	0	1	n/a	n/a	n/a
Deciduous cover	percent	9	8	0	45	11	24	54	45	38	24	60	n/a	n/a	n/a
Evergreen/ mixed cover	percent	2	1	n/a	1	1	0	16	21	38	57	26	n/a	n/a	n/a

Table 1. Site characteristics for the 14 Minnesota lakes that were sampled in this investigation.—Continued

[DOW, Division of Wildlife; km2, square kilometer; m, meter; m3, cubic meter;  $\mu$ g/L, microgram per liter; km, kilometer; n/a, not applicable; Eu; eutrophic; Meso, mesotrophic; Oligo, oligotrophic; POCIS, polar organic compound integrated samplers; --, not available; Date format: M/DD/YY]

	Unit	0wasso	Cedar	Budd	Red Sand	Sullivan	White Sand	Kabetogama	Shingobee	Stewart	Northern Light	Elk	Beast	Ryan	Bohall
Grassland cover	percent	0	0	4	4	11	2	2	18	0	0	0	n/a	n/a	n/a
Cropland cover	percent	n/a	n/a	76	2	49	2	0	2	n/a	n/a	0	n/a	n/a	n/a
Wetland cover	percent	3	2	2	14	7	18	10	2	1	4	1	n/a	n/a	n/a
Open water cover	percent	14	7	6	22	8	34	14	6	21	12	12	n/a	n/a	n/a
Barren cover	percent	n/a	n/a	0	n/a	n/a	n/a	0	n/a	n/a	0	n/a	n/a	n/a	n/a
Shrubland cover	percent	n/a	0	n/a	1	5	n/a	3	2	1	3	0	n/a	n/a	n/a
POCIS deployment	date	5/23/08	5/23/08	5/27/08	6/18/08	6/19/08	6/18/08	8/21/08	7/15/08	7/14/08	8/4/08	6/18/08	n/a	n/a	n/a
Water sampling	date	6/2/08	6/2/08	6/3/08	6/27/08	6/29/08	6/27/08	7/3/08	7/15/08	7/14/08	8/4/08	6/27/08	10/19/10	10/19/10	10/21/10

**Table 2.** Trace elements and major elements measured by inductively coupled plasma/mass spectrometry (Garbarino and Taylor, 1996; Taylor, 2001) and inductively coupled plasma/atomic-emission spectrometry (Garbarino and Taylor, 1979) at the U.S. Geological Survey National Research Program Laboratory, and the trace and major elements' approximate method detection limits (MDL).

[atomic symbols shown in parenthesis;  $\mu g/L$ , microgram per liter; mg/L, milligram per liter]

Element	MDL (µg/L)	Element	MDL (µg/L)
Aluminum (Al)	0.05	Manganese (Mn)	0.02
Antimony (Sb)	.02	Molybdenum (Mo)	.03
Arsenic (As)	.04	Neodymium (Nd)	.003
Barium (Ba)	.01	Nickel (Ni)	.02
Beryllium (Be)	.02	Potassium (K)	.02 mg/L
Bismuth (Bi)	.01	Praseodymium (Pr)	.005
Boron (B)	4	Rhenium (Re)	.001
Cadmium (Cd)	.006	Rubidium (Rb)	.002
Calcium (Ca)	.02 mg/L	Samarium (Sm)	.003
Cerium (Ce)	.001	Selenium (Se)	.2
Cesium (Cs)	.06	Silica (SiO2)	.1 mg/L
Chromium (Cr)	.2	Sodium (Na)	.02 mg/L
Cobalt (Co)	.01	Strontium (Sr)	.02
Copper (Cu)	.02	Sulfur (S)	.002 mg/L
Dysprosium (Dy)	.002	Tellurium (Te)	.01
Erbium (Er)	.002	Terbium (Tb)	.0003
Europium (Eu)	.001	Thallium (Tl)	.005
Gadolinium (Gd)	.003	Thorium (Th)	.004
Holmium (Ho)	.0005	Thulium (Tm)	.0005
Iron (Fe)	.7	Tungsten (W)	.004
Lanthanum (La)	.0005	Uranium (U)	.002
Lead (Pb)	.006	Vanadium (V)	.05
Lithium (Li)	.1	Ytterbium (Yb)	.0014
Lutetium (Lu)	.0005	Yttrium (Y)	.0004
Magnesium (Mg)	$.002~\mathrm{mg/L}$	Zinc (Zn)	.08
		Zirconium (Zr)	.01

**Table 3.** Acidic organic compounds measured by evaporation with derivatization and gas chromatography/mass spectrometry (Barber and others, 2000) at the U.S. Geological Survey National Research Program Laboratory.

[This report contains CAS Registry Numbers® (CASRN), which is a Registered Trademark of the American Chemical Society. CAS recommends the verification of the CASRNs through CAS Client ServicesSM; MDL, method detection limit;  $\mu$ g/L, microgram per liter; NA, not available; *, potential endocrine-disrupting compound]

Compound	CASRN	MDL (µg/L)
Ethylenediaminetetraacetic acid	60-00-4	0.05
Nitrilotriacetic acid	139-13-9	.05
4-Nonylphenolmonoethoxycarboxylic acid*	NA	.1
4-Nonylphenoldiethoxycarboxylic acid*	106807-78-7	.1
4-Nonylphenoltriethoxycarboxylic acid*	NA	.1
4-Nonylphenoltetraethoxycarboxylic acid*	NA	.1
Surrogate Standard		
4-normal-Nonylphenoldiethoxycarboxylate	NA	NA

**Table 4.** Neutral organic compounds measured by continuous liquid-liquid extraction using methylene chloride with gas chromatography/mass spectrometry (Barber and others, 2000) at the U.S. Geological Survey National Research Program Laboratory.

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Compound	CASRN	MDL (μg/L)
Bisphenol A*	80-05-7	0.01
2[3]-tert-Butyl-4-methylphenol	25013-16-5	.01
4-tert-Butylphenol	98-54-4	.01
Caffeine	58-08-2	.01
2,6-Di-tert-butyl-1,4-benzoquinone	719-22-2	.01
2,6-Di- <i>tert</i> -butyl-4-methylphenol	128-37-0	.01
2,6-Di- <i>tert</i> -butylphenol	128-39-2	.01
1,2-Dichlorobenzene	95-50-1	.01
1,3-Dichlorobenzene	541-73-1	.01
1,4-Dichlorobenzene*	106-46-7	.01
<i>N,N</i> -Diethyl- <i>meta</i> -toluamide	134-62-3	.01
4-Ethylphenol	123-07-9	.01
5-Methyl-1H-benzotriazole	136-85-6	.01
4-Methylphenol	106-44-5	.01
4-Nonylphenol *	25154-52-3	.05
4-Nonylphenolmonoethoxylate *	9016-45-9	.05
4-Nonylphenoldiethoxylate*	NA	.05
4-Nonylphenoltriethoxylate*	NA	.05
4-Nonylphenoltetraethoxylate*	NA	.05
4-normal-Octylphenol*	1806-26-4	.01
4-tert-Octylphenol*	140-66-9	.01
4-tert-Octylphenolmonoethoxylate*	9036-19-5	.01
4-tert-Octylphenoldiethoxylate*	NA	.01
4-tert-Octylphenoltriethoxylate*	NA	.01
4-tert-Octylphenoltetraethoxylate*	NA	.01
4-tert-Octylphenolpentaethoxylate*	NA	.01
4-tert-Pentylphenol*	80-46-6	.01
4-Propylphenol	645-56-7	.01
Triclosan	3380-34-5	.01
Surrogate Standards		
d6 Bisphenol A	86588-58-1	NA
d21 2,6-Di-tert-butyl-4-methylphenol	64502-99-4	NA
4-normal-Nonylphenol	104-40-5	NA
4-normal-Nonylphenolmonoethoxylate	NA	NA
4-normal-Nonylphenoldiethoxylate	NA	NA

**Figure 5.** Steroid and steroidal hormone compounds measured by octadecylsilica solid-phase extraction with derivatization and gas chromatography/tandem mass spectrometry (Barber and others, 2003; Foreman and others, 2012) at the U.S. Geological Survey National Research Program Laboratory.

[This report contains CAS Registry Numbers® (CASRN), which is a Registered Trademark of the American Chemical Society. CAS recommends the verification of the CASRNs through CAS Client ServicesSM; MDL, method detection limit;  $\mu$ g/L, microgram per liter; *, potential endocrine-disrupting compound; NA, not available; --, not applicable]

Compound	CASRN	MDL (μg/L)
4-Androstene-3,17-dione	63-05-8	0.0001
cis-Androsterone	53-41-8	.0001
Cholesterol	57-88-5	.005
Coprostanol	360-68-9	.005
Diethylstilbesterol*	56-53-1	.0001
Equilenin*	517-09-9	.0001
Equilin*	474-86-2	.0001
17α-Estradiol*	57-91-0	.0001
17β-Estradiol*	50-28-2	.0001
Estriol*	50-27-1	.0001
Estrone*	53-16-7	.0001
17α-Ethinylestradiol*	57-63-6	.0001
Mestranol*	72-33-3	.0001
19-Norethisterone*	68-22-4	.0001
Progesterone	57-83-0	.0001
Testosterone	58-22-0	.0001
dihydro-Testosterone	521-18-6	.0001
epi-Testosterone	481-30-1	.0001
11-keto-Testosterone	53187-98-7	.0001
Surrogate Standards		
d7-Androstenedione	67034-85-9	
d7-Cholesterol	83199-47-7	
d12-Chrysene	1719-03-5	
d4-Dihydrotestosterone	NA	
d4-17α-Ethinylestradiol	NA	
d4-17β-Estradiol	66789-03-5	
d3-Estriol	79037-36-8	
d4-Estrone	53866-34-5	
d4-Mestranol	NA	
d6-Norethindrone	NA	
d9-Progesterone	15775-74-3	
d5-Testosterone	21002-80-2	<b></b>

 Table 6.
 Summary of field measurements made at 11 Minnesota lakes during 2008.

 $[Date\ format:\ M/DD.\ \mu S/cm,\ microsiemen\ per\ centimeter,\ mg/L,\ milligram\ per\ liter;\ mg/L,\ std\ Unit,\ standard\ unit,\ mV,\ millivolts;\ NM,\ not\ measured]$ 

Waterbody	Date	Water temp °C	Specific conductivity µS/cm	Total dissolved solids mg/L	Salinity per thousands	Dissolved oxygen percent	Dissolved oxygen mg/L	pH std unit	Oxidation reduction potential mV
Cedar	5/23	18.1	580	430	0.33	120	11	8.2	140
Cedar	6/2	19.9	550	400	.3	140	13	8.5	150
Cedar	6/13	20.1	490	350	.26	100	9.5	7.5	140
Cedar	7/10	25.4	540	350	.26	83	6.6	8.8	130
Owasso Lake	5/23	16.6	330	260	.19	NM	10	7.6	150
Owasso Lake	6/2	20.0	330	280	.18	140	12	8.2	150
Owasso Lake	6/13	19.9	300	220	.16	89	8.1	7.3	140
Budd Lake	5/22	16.2	430	340	.25	96	9.4	10	120
Budd Lake	6/3	18.8	490	360	.27	NM	9.0	8.3	160
Budd Lake	6/12	20.5	420	300	.22	94	8.4	9.3	140
White Sand	7/9	26.2	140	86	.06	95	7.6	7.9	110
Red Sand Lake	6/18	24.1	53	37	.03	120	10	2.5	240
Red Sand Lake	7/9	26.9	170	100	.07	100	8.3	8.1	120
Sullivan Lake	6/18	21.8	89	60	.05	92	7.9	2.2	260
Elk Lake	6/18	18.2	220	160	.12	120	11	7.8	120
Elk Lake	7/9	21.8	250	180	.13	93	8.1	7.7	130
Stewart Lake	7/14	20.3	48	35	.02	96	8.5	7.8	68
Shingobee Lake	7/14	21.1	280	200	.14	85	7.6	7.6	140
Lake Kabetogama	7/2	18.6	67	50	.03	97	9.1	1.2	280
Northern Light Lake	8/3	21.8	33	25	.02	91	8.0	NM	110

**Table 7.** Summary of major- and trace-element data for water samples collected from 11 Minnesota lakes during 2008.  $[mg/L, milligram per liter; \mu g/L, microgram per liter; <, less than]$ 

Constituent	Unit	0wasso	Cedar	Budd	Sullivan	Red Sand	Shingobee	White Sand	Stewart	Kabetogama	Northern Light	Elk
Aluminum	μg/L	16	7.7	8.2	2.3	3.3	0.3	1.0	7.0	10	28	1.7
Antimony	$\mu \text{g/L}$	.39	.23	.22	.074	.033	.015	.042	.039	.048	.032	.021
Arsenic	μg/L	11	1.9	1.2	1.3	.93	.71	.63	.45	.38	.39	.91
Barium	μg/L	38	38	55	34	28	59	36	10	12	4.8	45
Beryllium	$\mu g/L$	.008	.006	<.005	<.005	.006	<.005	<.005	.005	.008	.016	<.005
Bismuth	$\mu g/L$	<.006	<.006	<.006	<.006	<.006	<.006	<.006	<.006	<.006	<.006	<.006
Boron	$\mu g/L$	22	20	33	26	16	26	16	14	7	8	27
Cadmium	$\mu g/L$	.055	.038	.015	<.002	.007	.009	.004	.011	.010	.015	.020
Cesium	$\mu g/L$	.05	.04	.03	.03	.03	.02	.04	.01	.04	.02	.03
Calcium	mg/L	25	36	57	38	15	46	25	14	11	4.5	34
Cerium	$\mu g/L$	.021	.011	.015	.0036	.0091	.0039	.0098	.018	.055	.18	.0038
Chromium	μg/L	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Cobalt	μg/L	.036	.051	.050	.032	.029	.0031	.016	.014	.048	.031	.016
Copper	μg/L	2.4	.81	2.5	.25	.37	.50	1.5	.93	.90	2.5	.19
Dysprosium	μg/L	.0024	.0017	.0025	.0007	.0014	.0003	.0008	.0037	.0057	.028	.0003
Erbium	μg/L	.0016	.0006	.0021	.0003	.0007	<.0001	.0007	.0019	.0037	.017	.0001
Europium	μg/L	.0013	.0009	<.0001	.0008	.0008	.0004	.0011	.0007	.0022	.0080	.0017
Gadolinium	μg/L	.0024	.0011	.0029	.0012	.0009	.0006	.0012	.0034	.0084	.039	.0005
Holmium	μg/L	.0004	.0003	.0004	.0002	.0003	.0001	.0002	.0006	.0014	.0059	.0001
Iron	μg/L	30	7	11	9	204	<2	112	33	23	376	137
Lanthanum	μg/L	.018	.0083	.0097	.0037	.0071	.0030	.0069	.014	.056	.20	.0026
Lead	μg/L	.60	.33	.20	.17	.12	.05	.07	.06	.12	.16	90
Lithium	μg/L	2.3	12	18	13	1.1	4.0	.85	1.0	1.1	.77	4.2
Lutetium	μg/L	.0002	.0002	.0004	.0001	.0004	.0000	.0003	.0003	.0004	.0026	.0001
Magnesium	mg/L	6.0	9.6	28	19	5.4	16	6.1	4.5	3.9	1.8	16
Manganese	μg/L	2.8	5.3	7.0	1.1	4.8	.26	1.0	1.1	2.1	5.4	3.3
Molybdenum	μg/L	0.63	0.91	2.6	0.43	0.04	0.38	0.08	0.20	0.18	0.17	0.36
Neodymium	μg/L	.018	.0069	.0097	.0020	.0051	.0021	.0050	.014	.045	.25	.0027
Nickel	μg/L	1.1	1.0	1.5	1.1	.37	.90	.60	.48	.70	.63	.67
Potassium	mg/L	3.6	2.9	2.3	4.0	.51	1.5	.40	.52	.89	.25	1.5
Praseodymium	μg/L	.0039	.0027	.0021	.0005	.0012	.0008	.0014	.0036	.011	.059	.0004

**Table 7.** Summary of major- and trace-element data for water samples collected from 11 Minnesota lakes during 2008.—Continued [mg/L, milligram per liter; μg/L, microgram per liter; <, less than]

Constituent	Unit	0wasso	Cedar	Budd	Sullivan	Red Sand	Shingobee	White Sand	Stewart	Kabetogama	Northern Light	Elk
Beryllium	μg/L	.008	.006	<.005	<.005	.006	<.005	<.005	.005	.008	.016	<.005
Rhenium	$\mu g/L$	.002	.003	.040	.012	.001	.001	.001	.002	.001	.001	<.001
Rubidium	$\mu g/L$	2.0	2.2	.89	2.4	.98	.98	.53	.55	1.2	.53	1.0
Samarium	$\mu g/L$	.0039	.0021	.0025	.0005	.0014	.0006	.0012	.0033	.0085	.046	.0005
Selenium	$\mu g/L$	.4	.2	1.4	.2	.2	<.1	.1	.1	<.1	.1	.3
Silica dioxide	mg/L	.10	.22	6.2	4.4	2.9	13	1.8	3.9	4.1	4.9	9.3
Sodium	mg/L	30	64	6.5	7.9	8.0	4.9	7.5	1.8	1.6	1.0	6.0
Strontium	$\mu g/L$	42	59	202	106	37	82	46	35	24	18	74
Sulfur	mg/L	1.7	4.3	14	2.5	.46	1.6	.48	1.1	1.4	1.1	.19
Tellerium	$\mu g/L$	<.007	<.007	.009	<.007	.007	<.007	<.007	<.007	<.007	<.007	<.007
Terbium	$\mu g/L$	.0005	.0003	.0003	.0001	.0002	.0001	.0001	.0004	.0011	.0050	.0000
Thallium	μg/L	.0044	.0038	.0066	.0031	.0031	.0010	.0040	.0023	.012	.0076	.0027
Thorium	μg/L	<.02	<.02	<.02	<.02	<.02	<.02	<.02	<.02	<.02	<.02	<.02
Thulium	μg/L	.0002	.0001	.0003	.0001	.0001	.0001	.0001	.0005	.0004	.0022	.0001
Tin	μg/L	.046	.013	.009	<.008	<.008	<.008	<.008	<.008	<.008	.025	.015
Tungsten	μg/L	.0061	.047	.012	.0023	.0047	.0021	<.001	.0018	.0093	<.001	<.001
Uranium	μg/L	.091	.34	8.9	.56	.011	.13	.018	.037	.060	.019	.021
Vanadium	μg/L	.47	.4	1.6	.16	.13	<.1	<.1	.19	.30	.40	<.1
Ytterbium	μg/L	.0011	.0009	.0018	.0006	.0008	<.0003	.0011	.0018	.0043	.016	<.0003
Yttrium	μg/L	.015	.0091	.020	.0048	.0072	.0031	.0066	.026	.041	.18	.0035
Zinc	μg/L	5.9	11	3.4	1.7	1.7	.92	1.3	7.9	11	28	14
Zirconium	μg/L	.05	.03	.13	.04	.02	.06	.04	.03	.06	.09	.03

 Table 8.
 Summary of acidic organic compound data for water samples collected from 11 Minnesota lakes during 2008.

[μg/L, microgram per liter; <, less than; Dup, duplicate sample]

Compound	Owasso µg/L	Cedar µg/L	Budd µg/L	Budd Dup µg/L	Sullivan µg/L	Red Sand µg/L	Shingobee µg/L	White Sand µg/L	Stewart µg/L	Kabetogama µg/L	Northern Light µg/L	Elk μg/L
Ethylenediaminetetraacetic acid	0.35	0.19	0.12	0.14	0.39	0.21	0.11	0.11	0.05	0.06	0.24	0.13
Nitrilotriacetic acid	<.05	.07	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
4-Nonylphenolmonoethoxycarboxylic acid	<.1	.27	<.1	<.1	<.1	<.1	.18	<.1	<.1	<.1	<.1	<.1
4-Nonylphenoldiethoxycarboxylic acid	<.1	.14	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1
4-Nonylphenoltriethoxycarboxylic acid	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1
4-Nonylphenoltetraethoxycarboxylic acid	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1	<.1
Surrogate Recovery (percent)												
4-normal-Nonylphenoldiethoxycarboxylic acid	44	49	36	30	32	39	39	38	40	38	32	43

Table 9. Summary of neutral organic compound data for water samples collected from 11 Minnesota lakes during 2008.

[avg, average;  $\mu$ g/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent]

Compound	Owasso µg/L	Cedar µg/L	Budd µg/L	Sullivan µg/L	Red Sand (avg) μg/L	Shingobee µg/L	White Sand μg/L	Stewart µg/L	Kabetogama (avg) µg/L	Northern Light (avg) µg/L	Elk (avg) µg/L
Bisphenol A	0.03	< 0.01	0.04	< 0.01	< 0.01	0.02	< 0.01	0.02	< 0.01	0.01	< 0.01
2[3]-tert-Butyl-4-methylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Butylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
Caffeine	.13	.09	.02	<.01	<.01	.01	<.01	.01	<.01	.02	.01
1,2-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
1,3-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
1,4-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
2,6-Di-tert-butyl-1,4-benzoquinone	<.01	.14	<.01	.08	.08	<.01	.09	<.01	<.01	<.01	.15
2,6-Di-tert-butyl-4-methylphenol	.07	.07	.07	<.01	.02	<.01	.02	<.01	<.01	.02	.02
2,6-Di-tert-butylphenol	<.01	<.01	.18	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
<i>N,N</i> -Diethyl- <i>meta</i> -toluamide	.09	.05	.03	.03	.01	.58	.03	.05	.02	.05	.22
4-Ethylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
5-Methyl-1H-benzotriazole	<.01	.04	.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-Methylphenol	.03	.02	<.01	.02	.02	.01	.02	.02	.02	.04	.01

**Table 9.** Summary of neutral organic compound data for water samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent]

Compound	Owasso µg/L	Cedar µg/L	Budd µg/L	Sullivan µg/L	Red Sand (avg) μg/L	Shingobee µg/L	White Sand μg/L	Stewart µg/L	Kabetogama (avg) µg/L	Northern Light (avg) µg/L	Elk (avg) μg/L
4-Nonylphenol	<.05	<.05	<.05	<.05	<.05	.11	<.05	<.05	<.05	.21	<.05
4-Nonylphenolmonoethoxylate	<.05	<.05	.06	<.05	<.05	.09	<.05	<.05	<.05	.17	<.05
4-Nonylphenoldiethoxylate	<.05	<.05	<.05	<.05	<.05	.17	<.05	<.05	<.05	.13	<.05
4-Nonylphenoltriethoxylate	<.05	<.05	<.05	<.05	<.05	.12	<.05	<.05	<.05	<.05	<.05
4-Nonylphenoltetraethoxylate	<.05	<.05	<.05	<.05	<.05	.03	<.05	<.05	<.05	<.05	<.05
4-normal-Octylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Octylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	.01	<.01
4-tert-Octylphenolmonoethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Octylphenoldiethoxylate	<.01	<.01	.03	<.01	<.01	.03	<.01	<.01	<.01	.04	<.01
4-tert-Octylphenoltriethoxylate	<.01	<.01	<.01	<.01	<.01	.01	<.01	<.01	<.01	<.01	<.01
4-tert-Octylphenoltetraethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Octylphenolpentaethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Pentylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-Propylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
Triclosan	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	.01	<.01
Surrogate Recovery (percent)											
d6-Bisphenol A	67	60	63	43	44	65	63	77	63	99	45
d21-2,6-Di- <i>tert</i> -butyl-4- methylphenol	27	24	7	10	26	36	25	42	40	55	18
4-normal-Nonylphenol	43	30	38	32	34	42	32	49	50	71	37
4-normal- Nonylphenolmonoethoxylate	22	17	24	28	21	36	20	31	34	46	27
4-normal-Nonylphenoldiethoxylate	17	13	22	21	17	24	14	14	22	22	20

**Table 9.** Summary of neutral organic compound data for water samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent]

Compound	Red Sand µg/L	Red Sand Dup µg/L	Elk µg/L	Elk Dup µg/L	Kabetogama µg/L	Kabetogama Dup µg/L	Northern Light µg/L	Northern Light Dup µg/L	Distilled Water Blank µg/L	Distilled Water Blank µg/L	Field Blank µg/L	Budd MS percent	White Sand MS percent
Bisphenol A	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01	0.01	< 0.01	< 0.01	< 0.01	34	72
2[3]-tert-Butyl-4-methylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	0	27
4-tert-Butylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	29	60
Caffeine	<.01	<.01	.01	<.01	<.01	<.01	.03	.01	<.01	.02	<.01	41	64
1,2-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	27	59
1,3-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	26	57
1,4-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	26	57
2,6-Di-tert-butyl-1,4-benzoquinone	.08	<.01	.15	<.01	<.01	<.01	<.01	<.01	.07	<.01	<.01	33	61
2,6-Di-tert-butyl-4-methylphenol	.01	.02	.02	.01	<.01	<.01	.02	<.01	.01	<.01	<.01	8	36
2,6-Di-tert-butylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	0	41
N,N-Diethyl-meta-toluamide	.01	.01	.27	.17	.02	.02	.07	.03	<.01	.08	<.01	49	73
4-Ethylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	29	59
5-Methyl-1H-benzotriazole	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	29	44
4-Methylphenol	.01	.02	.01	.02	.01	.03	.05	.03	<.01	<.01	<.01	26	57
4-Nonylphenol	<.05	<.05	<.05	<.05	<.05	<.05	.28	.15	<.05	<.05	<.05	15	59
4-Nonylphenolmonoethoxylate	<.05	<.05	<.05	<.05	<.05	<.05	.17	<.05	<.05	<.05	<.05	17	56
4-Nonylphenoldiethoxylate	<.05	<.05	<.05	<.05	<.05	<.05	<.05	.13	<.05	<.05	<.05	14	52
4-Nonylphenoltriethoxylate	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	12	43
4-Nonylphenoltetraethoxylate	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	10	35
4-normal-Octylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	18	58
4-tert-Octylphenol	<.01	<.01	<.01	<.01	<.01	<.01	.01	<.01	<.01	<.01	<.01	23	71
4-tert-Octylphenolmonoethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	25	67
4-tert-Octylphenoldiethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	.04	.04	<.01	<.01	<.01	17	56
4-tert-Octylphenoltriethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	14	44
4-tert-Octylphenoltetraethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	13	45
4-tert-Octylphenolpentaethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	9	28
4-tert-Pentylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	30	64

**Table 9.** Summary of neutral organic compound data for water samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent]

Compound	Red Sand µg/L	Red Sand Dup µg/L	Elk µg/L	Elk Dup µg/L	Kabetogama µg/L	Kabetogama Dup µg/L	Northern Light µg/L	Northern Light Dup µg/L	Distilled Water Blank µg/L	Distilled Water Blank µg/L	Field Blank µg/L	Budd MS percent	White Sand MS percent
4-Propylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	29	60
Triclosan	<.01	<.01	<.01	<.01	<.01	<.01	<.01	.01	<.01	<.01	<.01	20	62
Surrogate Recovery													
d6-Bisphenol A	37	51	41	50	47	79	112	87	13	61	117	45	66
d21-2,6-Di-tert-butyl-4-methylphenol	24	28	18	19	30	50	62	48	32	28	67	5	34
4-normal-Nonylphenol	31	36	34	40	38	61	79	63	48	91	120	17	52
4- <i>normal</i> -Nonylphenolmonoethoxylate	20	23	24	30	27	41	57	34	33	102	96	14	39
4-normal-Nonylphenoldiethoxylate	16	17	17	23	18	26	28	17	14	57	46	10	26

Table 10. Summary of steroid and steroidal hormone compound data for water samples collected from 11 Minnesota lakes during 2008.

 $[avg, average; \mu g/L, microgram\ per\ liter; <, less\ than; Dup, duplicate\ sample; MS, matrix\ spike\ recovery\ in\ percent]$ 

Compound	Owasso µg/L	Cedar μg/L	Budd μg/L	Sullivan µg/L	Red Sand (avg) µg/L	Shingobee µg/L	White Sand (avg) μg/L	Stewart µg/L	Kabetogama (avg) µg/L	Northern Light (avg) µg/L	Elk (avg) μg/L
4-Androstene-3,17-dione	< 0.0001	< 0.0001	< 0.0001	0.0007	0.0004	0.0005	0.0010	0.0003	0.0005	< 0.0001	0.0008
cis-Androsterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Cholesterol	9.0	7.8	2.5	7.1	5.7	<.005	7.5	<.005	2.0	<.005	.008
Coprostanol	NM	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005
Diethylstilbesterol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Equilenin	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Equilin	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0003	<.0001	<.0001	<.0001	<.0001
17α-Ethinylestradiol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
17α-Estradiol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
17β-Estradiol	.0001	.0004	<.0001	.0001	<.0001	<.0001	.0001	<.0001	<.0001	.0005	.0004
Estriol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Estrone	<.0001	<.0001	<.0001	.0007	.0006	.0011	.0006	.0015	<.0001	<.0001	.0011
Mestranol	.0002	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Norethindrone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Progesterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0005	<.0001	<.0001	<.0001	<.0001
dihydro-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
epi-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
11-keto-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Surrogate Recovery (percent	t)										
d7-Androstenedione	0	0	0	45	62	64	45	68	51	28	58
d7-Cholesterol	14	33	23	56	51	27	52	30	62	59	29
d12-Chrysene	79	92	82	88	84	76	86	84	98	89	83
d4-Dihydrotestosterone	0	0	0	62	86	79	28	81	31	27	55
d4-Ethinyl estradiol	49	60	57	82	81	53	84	74	91	86	41
d4-Estradiol	50	65	55	80	74	52	61	81	71	80	43
d3-Estriol	41	53	36	60	50	19	56	40	70	57	44
d4-Estrone	0	0	0	85	75	59	98	88	103	102	43
d4-Mestranol	56	63	59	89	93	64	89	79	90	85	40

**Table 10.** Summary of steroid and steroidal hormone compound data for water samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent]

Compound	Owasso µg/L	Cedar µg/L	Budd µg/L	Sullivan µg/L	Red Sand (avg) µg/L	Shingobee µg/L	White Sand (avg) µg/L	Stewart µg/L	Kabetogama (avg) µg/L	Northern Light (avg) µg/L	Elk (avg) μg/L
d6-Norethindrone	0	0	0	35	57	58	57	79	80	77	43
d9-Progesterone	0	0	0	0	2	19	0	19	1	4	1
d5-Testosterone	0	0	0	47	69	67	43	66	39	30	54

Duplicates, Blanks, and Matrix Spikes

Compound	Red Sand µg/L	Red Sand Dup µg/L	White Sand µg/L	White Sand Dup µg/L	Kabetogama μg/L	Kabetogama Dup μg/L	Northern Light µg/L	Northern Light Dup µg/L	Elk µg/L	Elk Dup µg/L	Field blank µg/L	Distilled Water Blank µg/L	Distilled Water Blank µg/L	Sullivan MS percent	Distilled Water MS percent
4-Androstene-3,17-dione	<.0001	.0004	.0010	.0010	.0006	.0004	<.0001	<.0001	.0008	.0009	<.0001	<.0001	<.0001	92	84
cis-Androsterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	99	108
Cholesterol	6.5	4.8	7.5	7.5	2.0	2.0	<.005	<.005	8.5	8.1	<.005	<.005	<.005	71	145
Coprostanol	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005	98	154
Diethylstilbesterol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	81	99
Equilenin	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	34	23
Equilin	<.0001	<.0001	<.0001	.0003	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	38	4
17α-Ethinylestradiol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	97	98
17α-Estradiol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	114	99
17β-Estradiol	<.0001	<.0001	.0001	.0001	<.0001	<.0001	.0008	.0001	.0004	.0004	<.0001	<.0001	<.0001	102	97
Estriol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	98	97
Estrone	<.0001	.0007	.0006	.0007	<.0001	<.0001	<.0001	<.0001	.0011	.0011	<.0001	<.0001	<.0001	90	79
Mestranol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	101	101
Norethindrone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	97	94

**Table 10.** Summary of steroid and steroidal hormone compound data for water samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent]

Compound	Red Sand µg/L	Red Sand Dup µg/L	White Sand µg/L	White Sand Dup µg/L	Kabetogama µg/L	Kabetogama Dup µg/L	Northern Light µg/L	Northern Light Dup µg/L	Elk µg/L	Elk Dup µg/L	Field blank µg/L	Distilled Water Blank µg/L	Distilled Water Blank µg/L	Sullivan MS percent	Distilled Water MS percent
Progesterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	0	88
Testosterone	<.0001	<.0001	<.0001	.0005	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	92	88
dihydro-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001		
epi-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	108	92
11-keto-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	52	89
Surrogate Recovery (pe	ercent)														
d7-Androstenedione	66	58	46	43	51	51	28	28	38	79	112	123	84	49	104
d7-Cholesterol	56	46	62	43	58	67	96	22	13	45	12	19	11	31	8
d12-Chrysene	88	79	92	80	99	97	94	85	74	92	95	99	118	76	87
d4-Dihydrotestosterone	77	94	31	25	31	31	29	25	36	73	85	80	76	67	70
d4-Ethinyl estradiol	85	77	91	77	91	91	104	68	29	54	82	74	73	63	51
d4-Estradiol	76	72	65	57	71	72	94	66	32	55	88	65	66	62	48
d3-Estriol	53	48	66	47	69	70	74	40	36	52	50	50	47	38	33
d4-Estrone	83	68	104	93	103	102	117	87	33	52	98	81	79	77	64
d4-Mestranol	99	87	93	84	90	91	102	69	29	51	89	88	75	80	66
d6-Norethindrone	53	61	62	53	78	82	82	71	23	62	91	108	94	46	83
d9-Progesterone	1	3	0	1	1	2	1	8	0	2	28	100	88	1	58
d5-Testosterone	63	75	47	39	37	41	31	29	36	72	97	103	78	54	84

**Table 11.** Summary of neutral organic compound data for sediment samples collected from 11 Minnesota lakes during 2008. [avg, average; μg/kg, microgram per kilogram; <, less than; MS, matrix spike recovery in percent; Dup, duplicate sample; Trip, triplicate sample]

Compound	Owasso µg/kg	Cedar (avg) µg/kg	Budd µg/kg	Sullivan µg/kg	Red Sand (avg) µg/kg	Shingobee (avg) µg/kg	White Sand µg/kg	Stewart μg/kg	Kabe- togama µg/kg	Northern Light µg/kg	Elk µg/kg
Bisphenol A	20	6.3	17	6.7	17	19	35	<1	16	3.1	7.7
2[3]-tert-Butyl-4-methylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-tert-Butylphenol	<1	<1	<1	6	<1	<1	<1	<1	<1	<1	<1
Caffeine	<1	110	<1	<1	<1	<1	<1	<1	<1	<1	<1
1,2-Dichlorobenzene	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
1,3-Dichlorobenzene	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
1,4-Dichlorobenzene	<1	<1	<1	<1	1.1	0	<1	<1	<1	1	<1
2,6-Di-tert-butyl-1,4-benzoquinone	< 200	< 200	< 200	< 200	< 200	< 200	< 200	< 200	< 200	< 200	< 200
2,6-Di-tert-butyl-4-methylphenol	<3	<3	<3	<3	<3	<3	<3	<3	<3	4	<3
2,6-Di-tert-butylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
<i>N,N</i> -Diethyl- <i>meta</i> -toluamide	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-Ethylphenol	59	8.2	<3	21	<3	96	23	<3	607	34	18
5-methyl-1H-Benzotriazole	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-Methylphenol	595	12	<3	131	355	160	186	<3	1,100	128	34
4-Nonylphenol	<100	<100	<100	<100	100	350	<100	<100	<100	<100	<100
4-Nonylphenolmonoethoxylate	<20	<20	<20	< 20	<20	30	<20	<20	<20	<20	< 20
4-Nonylphenoldiethoxylate	<1	<1	<1	<1	<1	59	<1	<1	<1	<1	<1
4-Nonylphenoltriethoxylate	<1	<1	<1	62	<1	<1	<1	<1	<1	<1	<1
4-Nonylphenoltetraethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-normal-Octylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-tert-Octylphenol	98	4.4	<3	8	<3	22	33	<3	<3	<3	5
4-tert-Octylphenolmonoethoxylate	<1	<1	<1	<1	1.3	6	<1	<1	<1	<1	<1
4-tert-Octylphenoldiethoxylate	<1	<1	<1	<1	21	<1	74	3	45	<1	<1
4-tert-Octylphenoltriethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-tert-Octylphenoltetraethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-tert-Octylphenolpentaethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
4-tert-Pentylphenol	88	<1	<1	23	<1	<1	74	<1	<1	<1	<1
4-Propylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Triclosan	33	44	<1	<1	<1	<1	<1	<1	<1	<1	<1

**Surrogate Recovery (percent)** 

**Table 11.** Summary of neutral organic compound data for sediment samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/kg, microgram per kilogram; <, less than; MS, matrix spike recovery in percent; Dup, duplicate sample; Trip, triplicate sample]

Compound	Owasso µg/kg	Cedar (avg) µg/kg	Budd µg/kg	Sullivan µg/kg	Red Sand (avg) µg/kg	Shingobee (avg) µg/kg	White Sand µg/kg	Stewart μg/kg	Kabe- togama µg/kg	Northern Light µg/kg	Elk μg/kg
d6-Bisphenol A	59	51	54	48	75	61	87	25	54	99	55
d21-2,6-Di-tert-butyl-4-methylphenol	19	26	4	5	48	24	95	3	17	53	4
4-normal-Nonylphenol	114	108	140	97	133	105	144	45	92	148	117
4-normal-Nonylphenolmonoethoxylate	216	73	229	138	163	137	193	66	183	180	168
4-normal-Nonylphenoldiethoxylate	106	66	214	90	101	109	122	43	139	174	48

Replicates, Blanks, and Matrix Spikes

Compound	Cedar µg/kg	Cedar Dup µg/kg	Cedar Trip µg/kg	Red Sand µg/kg	Red Sand Dup µg/kg	Shin- gobee µg/kg	Shingobee Dup µg/kg	Sand Blank µg/kg	Sand Blank µg/kg	Sand Blank µg/kg	Red Sand MS percent	Red Sand MS Dup percent
Bisphenol A	<1	<1	6	5	28	14	24	<1	<1	<1	119	95
2[3]-tert-Butyl-4-methylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	77	91
4-tert-Butylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	178	150
Caffeine	<1	53	161	<1	<1	<1	<1	<1	<1	<1	0	0
1,2-Dichlorobenzene	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	8	7
1,3-Dichlorobenzene	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	4	3
1,4-Dichlorobenzene	<1	<1	<1	1	<1	2	<1	1	<1	<1	5	4
2,6-Di-tert-butyl-1,4-benzoquinone	< 200	< 200	< 200	< 200	< 200	< 200	< 200	< 200	< 200	< 200	28	25
2,6-Di-tert-butyl-4-methylphenol	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	107	90
2,6-Di-tert-butylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	89	79
N,N-Diethyl-meta-toluamide	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	18	72
4-Ethylphenol	10	<3	7	<3	<3	70	122	<3	<3	<3	78	126
5-methyl-1H-Benzotriazole	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	4	10

**Table 11.** Summary of neutral organic compound data for sediment samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/kg, microgram per kilogram; <, less than; MS, matrix spike recovery in percent; Dup, duplicate sample; Trip, triplicate sample]

Compound	Cedar µg/kg	Cedar Dup µg/kg	Cedar Trip µg/kg	Red Sand µg/kg	Red Sand Dup µg/kg	Shin- gobee µg/kg	Shingobee Dup µg/kg	Sand Blank µg/kg	Sand Blank µg/kg	Sand Blank µg/kg	Red Sand MS percent	Red Sand MS Dup percent
4-Methylphenol	<3	6	18	31	680	108	213	<3	<3	<3	14	122
4-Nonylphenol	<100	<100	<100	103	<100	<100	349	<100	<100	<100	208	168
4-Nonylphenolmonoethoxylate	< 20	< 20	<20	< 20	<20	30	<20	< 20	<20	< 20	153	124
4-Nonylphenoldiethoxylate	<1	<1	<1	<1	<1	59	<1	<1	<1	<1	119	114
4-Nonylphenoltriethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	130	115
4-Nonylphenoltetraethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	33	32
4-normal-Octylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	241	193
4-tert-Octylphenol	<3	<3	4	<3	<3	22	<3	<3	<3	<3	216	183
4-tert-Octylphenolmonoethoxylate	<1	<1	<1	<1	1	<1	6	<1	<1	<1	207	174
4-tert-Octylphenoldiethoxylate	<1	<1	<1	21	<1	<1	<1	<1	<1	<1	105	95
4-tert-Octylphenoltriethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	108	100
4-tert-Octylphenoltetraethoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	173	151
4-tert-Octylphenolpentathoxylate	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	0	0
4-tert-Pentylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	190	150
4-Propylphenol	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	176	152
Triclosan	<1	<1	44	<1	<1	<1	<1	<1	<1	<1	184	156
Surrogate Recovery (percent)												
d6-Bisphenol A	44	44	65	91	59	58	63	68	46	58	110	65
d21-2,6-Di-tert-butyl-4-methylphenol	29	22	29	39	56	29	19	64	33	60	86	59
4-normal-Nonylphenol	85	121	117	136	129	99	110	105	117	142	199	119
4-normal-Nonylphenolmonoethoxylate	0	111	106	173	152	117	156	70	120	46	343	220
4-normal-Nonylphenoldiethoxylate	49	80	68	123	80	91	127	52	72	32	113	77

**Table 12.** Summary of steroid and steroidal hormone compound data for sediment samples collected from 11 Minnesota lakes during 2008. [avg, average; μg/kg, microgram per kilogram; <, less than; Dup, duplicate sample; Trip, triplicate sample; MS, matrix spike recovery in percent]

Compound	Owasso µg/kg	Cedar (avg) µg/kg	Budd µg/kg	Sullivan µg/kg	Red Sand (avg) µg/kg	Shingobee (avg) µg/kg	White Sand µg/kg	Stewart µg/kg	Kabetogama µg/kg	Northern Light µg/kg	Elk µg/kg
4-Androstene-3,17-dione	2.3	< 0.05	0.22	0.39	0.55	0.94	0.59	0.33	< 0.05	0.61	0.47
cis-Androsterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Cholesterol	4,200	330	530	1,400	1,200	1,600	770	1,200	3,600	320	1,800
Coprostanol	790	47	32	360	320	340	35	130	380	36	230
Diethylstilbesterol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	3.8	.36	<.05
Equilenin	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Equilin	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
17α-Estradiol	<.05	<.05	.08	.06	.10	.17	.46	.08	<.05	<.05	<.05
17β-Estradiol	1.3	<.05	.05	.15	.35	1.3	.58	.12	.85	.08	.23
Estriol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Estrone	2.9	<.05	.27	.82	.69	2.4	.99	.59	5.6	.32	3.1
17α-Ethinylestradiol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Mestranol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Norethindrone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Progesterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Testosterone	1.4	<.05	<.05	<.05	.26	<.05	.23	<.05	2.7	<.05	<.05
dihydro-Testosterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
epi-Testosterone	<.05	<.05	<.05	<.05	.14	<.05	<.05	<.05	.89	<.05	<.05
11-keto-Testosterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05
Surrogate Recovery (per	cent)										
d7-Androstenedione	19	0	33	13	15	28	26	10	51	37	11
d7-Cholesterol	7	1	9	11	20	12	13	4	39	16	8
d8-Diethylstilbesterol	25	0	25	64	54	43	50	22	24	26	64
d4-Dihydrotestosterone	24	2	53	16	26	43	46	15	56	58	27
d4-Ethinylestradiol	41	3	40	37	62	63	68	33	92	64	59
d4-Estradiol	25	2	34	47	53	54	58	22	33	20	38
d4-Mestranol	47	3	75	39	63	83	2	38	94	71	80
d6-Norethindrone	11	0	21	8	17	16	18	5	27	23	8
d5-Testosterone	23	1	33	15	21	32	32	10	51	38	14

## Replicates, Blanks, and Matrix Spikes

**Table 12.** Summary of steroid and steroidal hormone compound data for sediment samples collected from 11 Minnesota lakes during 2008.—Continued [avg, average; μg/kg, microgram per kilogram; <, less than; Dup, duplicate sample; Trip, triplicate sample; MS, matrix spike recovery in percent]

Compound	Cedar µg/kg	Cedar Dup µg/kg	Cedar Trip µg/kg	Red Sand µg/kg	Red Sand Dup µg/kg	Shingobee µg/kg	Shingobee Dup µg/kg	Sand Blank µg/kg	Sand Blank µg/kg	Sand Blank µg/kg	Red Sand MS μg/kg	Red Sand MS Dup µg/kg
4-Androstene-3,17-dione	<.05	<.05	<.05	<.05	.55	.97	.92	<.05	.11	.06	25	94
cis-Androsterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	86	100
Cholesterol	420	320	260	870	1400	910	2400	41	72	12	52	90
Coprostanol	50	51	41	270	370	220	460	<5	<5	<5	30	44
Diethylstilbesterol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	71	120
Equilenin	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	87	92
Equilin	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	17	19
17α-Estradiol	<.05	<.05	<.05	.07	.13	.16	.17	<.05	<.05	<.05	57	68
17β-Estradiol	<.05	<.05	<.05	.20	.49	1.00	1.58	<.05	<.05	<.05	53	66
Estriol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	0	77
Estrone	<.05	<.05	<.05	.30	1.08	1.98	2.78	<.05	<.05	<.05	43	54
17α-Ethinylestradiol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	77	77
Mestranol	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	60	78
Norethindrone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	0	139
Progesterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	0	0
Testosterone	<.05	<.05	<.05	<.05	.26	<.05	<.05	<.05	<.05	<.05	118	104
dihydro-Testosterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05		
epi-Testosterone	<.05	<.05	<.05	<.05	.14	<.05	<.05	<.05	<.05	<.05	126	106
11-keto-Testosterone	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	<.05	0	0
Surrogate Recovery (perc	ent)											
d7-Androstenedione	1	0	0	6	25	32	24	19	12	18	5	20
d7-Cholesterol	1	0	1	29	12	12	11	10	14	33	45	27
d8-Diethylstilbesterol	0	0	0	56	52	39	46	32	0	65	77	62
d4-Dihydrotestosterone	2	0	2	14	38	52	34	63	50	69	35	46
d4-Ethinyl estradiol	3	1	4	65	60	64	62	54	20	67	95	83
d4-Estradiol	3	1	3	55	52	49	60	60	29	126	84	53
d4-Mestranol	4	1	5	57	69	95	72	64	51	72	88	74
d6-Norethindrone	0	0	0	2	32	19	13	15	10	18	0	7
d5-Testosterone	1	0	1	6	35	36	28	31	22	33	6	34

**Table 13.** Summary of neutral organic compound data for polar organic compound integrative samplers (POCIS) deployed in 11 Minnesota lakes during 2008. [µg/POCIS, microgram per POCIS; <, less than]

Compound	Owasso μg/ POCIS	Cedar µg/ POCIS	Budd µg/ POCIS	Sullivan µg/ POCIS	Sullivan Day 7 µg/ POCIS	Sullivan Day 14 µg/ POCIS	Sullivan Day 21 µg/ POCIS	Red Sand µg/ POCIS	Shingobee µg/ POCIS	White Sand µg/ POCIS	Stewart µg/ POCIS	Kabetogama µg/ POCIS	Northern Light µg/ POCIS	Elk μg/ POCIS
Bisphenol A	<0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	<0.01	< 0.01
2[3]-tert-Butyl-4-methylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Butylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
Caffeine	.02	.11	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
1,2-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
1,3-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
1,4-Dichlorobenzene	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
2,6-Di- <i>tert</i> -butyl-1,4-benzoquinone	<.01	<.01	<.01	.05	.07	.08	.05	.04	.29	.02	.21	.18	.08	.06
2,6-Di- <i>tert</i> -butyl-4-methylphenol	.35	<.01	<.01	.22	.22	.24	<.01	.17	.02	.06	.01	.16	.18	.18
2,6-Di- <i>tert</i> -butylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
N,N-Diethyl-meta-toluamide	.12	.09	<.01	.08	.02	.02	.04	.03	.01	.09	.06	.02	.04	.03
4-Ethylphenol	.02	.04	<.01	<.01	<.01	<.01	.01	<.01	.01	<.01	.01	.01	<.01	.03
5-methyl-1H-Benzotriazole	<.01	.06	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-Methylphenol	.06	.03	.02	.06	.01	.01	.02	.04	.06	.01	.06	.26	.04	.03
4-Nonylphenol	.09	.06	.03	.09	.05	.04	.06	.12	.06	.08	.09	.05	.06	.05
4-Nonylphenolmonoethoxylate	<.01	<.01	<.01	.04	<.01	<.01	.03	.03	<.01	.02	<.01	<.01	<.01	<.01
4-Nonylphenoldiethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	.12	<.01	<.01	<.01	<.01	<.01	<.01
4-Nonylphenoltriethoxylate	<.01	<.01	<.01	.06	<.01	<.01	<.01	<.01	<.01	.09	<.01	<.01	<.01	<.01
4-Nonylphenoltetraethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-normal-Octylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Octylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Octylphenolmonoethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4- <i>tert</i> -Octylphenoldiethoxylate	<.01	<.01	<.01	<.01	.02	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4- <i>tert</i> -Octylphenoltriethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4- <i>tert</i> -Octylphenoltetraethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-tert-Octylphenolpentaethoxylate	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4- <i>tert</i> -Pentylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
4-Propylphenol	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
Triclosan	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
Surrogate Recovery (percent)														
d6-Bisphenol A	22	20	9	147	167	92	107	123	161	131	144	99	125	123
d21-2,6-Di- <i>tert</i> -butyl-4-methylphenol	13	3	6	23	36	36	8	8	73	3	40	48	17	70
4- <i>normal</i> -Nonylphenol	23	17	8	144	146	50	106	116	143	116	133	101	115	99
4- <i>normal</i> -Nonylphenolmonoethoxylate	18	13	6	197	187	53	130	155	189	158	170	131	144	128
4- <i>normal</i> -Nonylphenoldiethoxylate	96	0	24	685	1250	261	863	95	1276	74	92	857	136	90

**Table 14.** Summary of steroid and steroidal hormone compound data for passive organic compound integrative samplers (POCIS) deployed in 11 Minnesota lakes during 2008.—Continued

[avg, average; µg/POCIS, microgram per POCIS; <, less than; Dup, duplicate sample; Trip, triplicate sample; --, not quantified]

Compound	Owasso (avg) µg/ POCIS	Cedar (avg) µg/ POCIS	Budd (avg) µg/ POCIS	Sullivan µg/ POCIS	Sullivan Day 7 µg/ POCIS	Sullivan Day 14 µg/ POCIS	Sullivan Day 21 µg/ POCIS	Red Sand μg/ POCIS	Shingobee µg/ POCIS	White Sand µg/ POCIS	Stewart μg/ POCIS	Kabetogama µg/ POCIS	Northern Light µg/ POCIS	Elk μg/ POCIS
POCIS extracted	1	1	1	3	3	3	3	3	3	3	3	3	3	3
4-Androstene-3,17-dione	<.0001	<.0001	.0023	.0005	<.0001	<.0001	<.0001	.0007	<.0001	.0009	.0007	<.0001	<.0001	.0008
cis-Androsterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Cholesterol	<.5	<.5	4.6	.5	<.5	<.5	<.5	.5	<.5	<.5	<.5	<.5	<.5	<.5
Coprostanol	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Diethylstilbesterol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Equilenin	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Equilin	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
17α-Estradiol	<.0001	<.0001	<.0001	.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0002
17β-Estradiol	.0005	.0006	.0003	.0003	<.0001	.0001	<.0001	<.0001	.0001	.0001	.0002	.0001	<.0001	.0003
Estriol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Estrone	.0012	.0012	.0010	.0013	<.0001	<.0001	.0004	.0012	.0008	.0011	.0022	<.0001	<.0001	.0010
17α-Ethinylestradiol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Mestranol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Norethindrone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Progesterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Testosterone	.0004	<.0001	.0004	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0002	<.0001
dihydro-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
epi-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0002
11-keto-Testosterone	<.0001	<.0001	.0017	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Surrogate Recovery (perc	ent)													
d7-Androstenedione	136	132	143	149	109	75	81	93	91	54	107	79	87	80
d7-Cholesterol	59	63	83	45	81	23	35	68	28	23	65	30	43	20
d8-Diethylstilbestrol	94	84	91	101	60	37	38	55	68	55	69	45	65	55
d4-Ethinyl estradiol	109	106	124	143	100	65	65	88	68	59	98	57	74	56
d4-Estradiol	95	88	104	1	139	88	74	111	93	74	108	78	76	80
d3-Estriol														
d4-Estrone	124	118	138	140	106	69	76	89	82	54	95	67	82	65
d4-Mestranol	89	90	100	118	90	41	59	70	64	49	78	55	65	50
d6-Norethindrone	128	124	138	160	110	86	75	94	78	58	104	74	79	67
d9-Progesterone	170	166	194	200	139	75	81	135	75	81	144	92	103	58
d5-Testosterone	109	109	125	140	92	63	59	78	67	56	89	62	67	65

## Replicates

**Table 14.** Summary of steroid and steroidal hormone compound data for passive organic compound integrative samplers (POCIS) deployed in 11 Minnesota lakes during 2008.—Continued

 $[avg, average; \mu g/POCIS, microgram\ per\ POCIS; <, less\ than; Dup, duplicate\ sample; Trip, triplicate\ sample; --, not\ quantified]$ 

	0wasso	Owasso Dup	Owasso Trip	Cedar	Cedar Dup	Cedar Trip	Budd	Budd Dup	Budd Trip
Compound	μg/ POCIS								
POCIS extracted	1	1	1	1	1	1	1	1	1
4-Androstene-3,17-dione	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0020	.0022	.0028
cis-Androsterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Cholesterol	<.5	<.5	<.5	<.5	<.5	<.5	3.4	5.1	5.4
Coprostanol	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Diethylstilbesterol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Equilenin	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Equilin	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
17α-Estradiol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
17β-Estradiol	.0004	.0002	.0007	.0010	.0004	.0005	.0002	<.0001	.0005
Estriol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Estrone	.0014	.0010	.0014	.0011	.0014	.0011	.0010	<.0001	.0011
17α-Ethinylestradiol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Mestranol	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Norethindrone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Progesterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Testosterone	<.0001	<.0001	.0004	<.0001	<.0001	<.0001	<.0001	.0003	.0004
dihydro-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
epi-Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
11- <i>keto</i> -Testosterone	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	.0017	<.0001	<.0001
Surrogate Recovery (perc	ent)								
d7-Androstenedione	87	160	160	85	139	173	107	153	168
d7-Cholesterol	49	56	73	16	90	83	67	94	88
d8-Diethylstilbestrol	46	119	118	62	82	108	62	100	113
d4-Ethinyl estradiol	78	120	129	54	121	144	97	137	139
d4-Estradiol	64	109	112	52	99	112	89	103	120
d3-Estriol	0	0	0	0	0	0	0	0	0
d4-Estrone	75	151	147	70	126	159	103	153	157
d4-Mestranol	62	100	106	54	98	118	77	112	113
d6-Norethindrone	88	143	153	69	137	166	107	151	155
d9-Progesterone	114	189	206	66	205	228	139	226	219
d5-Testosterone	72	122	133	66	115	145	93	134	147

 Table 15.
 Summary of field measurements for water samples collected from four Minnesota lakes during 2010.

 $[^{\circ}C, degrees \ Celsius; \mu S/cm, microsiemens \ per \ centimeter; \ mg/L, \ milligrams \ per \ liter; \ std \ unit, \ standard \ unit]$ 

	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
Elk	11.8	294	8.6	8.4
Beast	10.7	23	9.5	6.9
Bohall	9.6	182	10.0	8.0
Ryan	10.7	23	9.8	6.8

**Table 16.** Summary of acidic organic compound data for water samples collected from four Minnesota lakes during 2010.

[µg/L, microgram per liter; <, less than]

Compound	Elk μg/L	Beast μg/L	Bohall μg/L	Ryan μg/L
Ethylenediaminetetraacetic acid	0.53	< 0.05	0.06	0.40
Nitrilotriacetic acid	< .05	< .05	< .05	< .05
4-Nonylphenolmonoethoxycarboxylic acid	< .1	< .1	< .1	< .1
4-Nonylphenoldiethoxycarboxylic acid	< .1	< .1	< .1	< .1
4-Nonylphenoltriethoxycarboxylic acid	< .1	< .1	< .1	< .1
4-Nonylphenoltetraethoxycarboxylic acid	< .1	< .1	< .1	< .1
Surrogate Recovery (percent)				
4-normal-Nonylphenoldiethoxycarboxylic acid	61	59	67	54

**Table 17.** Summary of neutral organic compound data for water samples collected from four Minnesota lakes during 2010. [μg/L, microgram per liter; <, less than; --, not quantifiable]

Compound	Elk µg/L	Beast μg/L	Bohall µg/L	Ryan µg/L
Acetylhexamethyltetrahydronaphthalene	< 0.01	< 0.01	< 0.01	<0.01
Bisphenol A	< .01	< .01	< .01	< .01
2[3]-tert-Butyl-4-methylphenol	< .01	< .01	< .01	< .01
4- <i>tert</i> -Butylphenol	< .01	< .01	< .01	< .01
Caffeine	< .01	< .01	< .01	< .01
2,6-Di-tert-butyl-1,4-benzoquinone	.05	.04	.02	< .01
2,6-Di-tert-butyl-4-methylphenol	.01	< .01	< .01	< .01
2,6-Di- <i>tert</i> -butylphenol	< .01	< .01	< .01	< .01
1,2-Dichlorobenzene	< .01	< .01	< .01	< .01
1,3-Dichlorobenzene	< .01	< .01	< .01	< .01
1,4-Dichlorobenzene	< .01	< .01	< .01	< .01
<i>N,N</i> -Diethyl- <i>meta</i> -toluamide	< .01	< .01	< .01	< .01
4-Ethylphenol	< .01	< .01	< .01	< .01
Hexahydrohexamethylcyclopentabenzopyran	< .01	< .01	< .01	< .01
5-Methyl-1H-benzotriazole	< .01	< .01	< .01	< .01
4-Methylphenol	< .01	.01	< .01	.01
4-Nonylphenol	< .05	< .05	< .05	< .05
4-Nonylphenolmonoethoxylate	< .05	< .05	< .05	< .05
4-Nonylphenoldiethoxylate	< .05	< .05	< .05	< .05
4-normal-Octylphenol	< .01	< .01	< .01	< .01
4- <i>tert</i> -Octylphenol	< .01	< .01	< .01	< .01
4- <i>tert</i> -Octylphenolmonoethoxylate	< .01	< .01	< .01	< .01
4- <i>tert</i> -Octylphenoldiethoxylate	< .01	< .01	< .01	< .01
4- <i>tert</i> -Octylphenoltriethoxylate	< .01	< .01	< .01	< .01
4- <i>tert</i> -Octylphenoltetraethoxylate	< .01	< .01	< .01	< .01
4- <i>tert</i> -Octylphenolpentaethoxylate	< .01	< .01	< .01	< .01
4- <i>tert</i> -Pentylphenol	< .01	< .01	< .01	< .01
4-Propylphenol	< .01	< .01	< .01	< .01
Triclosan	< .01	< .01	< .01	< .01
Surrogate Recovery (percent)				
d6-Bisphenol A	27	49	52	32
d21-2,6-Di- <i>tert</i> -butyl-4-methylphenol	53	85	21	63
4-normal-Nonylphenol	38	56	10	37
4-normal-Nonylphenolmonoethoxylate	38	51	47	37
4-normal-Nonylphenoldiethoxylate	46	63		34

**Table 18.** Summary of steroid and steroidal hormone compound data for water samples collected from four Minnesota lakes during 2010.

 $[\mu g/L,\,microgram\,per\,liter;\,<,\,less\,than]$ 

Compound	Elk μg/L	Beast μg/L	Bohall μg/L	Ryan µg/L
4-Androstene-3,17-dione	< 0.0001	< 0.0001	< 0.0001	< .0001
cis-Androsterone	< .0001	< .0001	< .0001	< .0001
Cholesterol	1.4	1.2	1.2	2.4
Coprostanol	.06	.08	.01	< .005
Diethylstilbestrol	< .0001	< .0001	< .0001	< .0001
Equilenin	< .0001	< .0001	< .0001	< .0001
Equilin	< .0001	< .0001	< .0001	< .0001
17α-Estradiol	< .0001	< .0001	< .0001	< .0001
17β-Estradiol	< .0001	< .0001	< .0001	< .0001
Estriol	< .0001	< .0001	< .0001	< .0001
Estrone	.0001	.0001	.0001	.0008
17α-Ethynylestradiol	< .0001	< .0001	< .0001	< .0001
Mestranol	< .0001	< .0001	< .0001	< .0001
Norethindrone	< .0001	< .0001	< .0001	< .0001
Progesterone	< .0001	< .0001	< .0001	< .0001
Testosterone	< .0001	< .0001	< .0001	< .0001
dihydro-Testosterone	< .0001	< .0001	< .0001	< .0001
epi-Testosterone	.0001	< .0001	< .0001	< .0001
11-keto-Testosterone	< .0001	< .0001	< .0001	< .0001
Surrogate Recovery (percent)				
d7-Cholesterol	64	40	48	54
d12-Chrysene	101	100	99	98
d8-Diethylstibestrol	75	78	83	90
¹³ C ₆ -Estradiol	57	60	76	82
d2-epi-Estriol	96	90	66	94
³ C ₆ -Estrone	115	109	95	105
d4-Ethynylestradiol	94	90	83	91
d3-Medroxyprogesterone	76	67	56	37
d4-Mestranol	95	90	82	90
d3-Nandrolone	102	93	86	83

 Table 19.
 Summary of field data for preliminary microhabitat selection at Sullivan Lake, Minn., June 1, 2010.

[Std unit, standard unit;  $\mu$ S/cm, microsiemens per centimeter; Temp, temperature; °C, degrees Celsius; mg/L, milligrams per liter; NM, not measured; Temp residual = Temp lake minus Temp pore; Temp residual less than 2°C indicates lake water loss downward to groundwater; Temp residual from 2°C to 5°C indicates mixing of groundwater and surface water; Temp residual greater than 5 indicates groundwater discharge to lake]

Location	Study site	Matrix	Lakebed	pH std unit	Specific conductance µS/cm	Nitrate mg/L	Temp °C	Temp residual	Assumed water flow
		Pore	Silt/clay	NM	1,395	0.2	18.6–19.1	3.4-3.9	Mixing
	Southwest	Lake	Silt/clay	7.16	416	.2	22.5		
	of site B	Pore	Silt/clay	NM	548	.2	16.0-16.9	5.6-6.5	Into Lake
		Lake	Silt/clay	7.89	379	.2	22.5		
		Pore	Sandy	NM	590	NM	16.6	7.1	Into Lake
		Lake	Sandy	7.87	403	NM	23.7		
		Pore	Sandy	NM	610	.1	21.4	2.3	Mixing
Surface water		Lake	Sandy	8.19	402	NM	23.7		
and pore water		Pore	Sandy	NM	754	NM	NM	NM	
physio-chemical	Site B	Lake	Sandy	7.85	424	NM	23.7		
characteristics of the		Pore	Sandy	NM	721	NM	NM	NM	
eastern shoreline		Pore	Sandy	NM	871	NM	NM	NM	
of Sullivan Lake		Pore	Sandy	NM	916	NM	NM	NM	
(from southwest		Lake	Sandy	7.75	400	.2	24.39		
to northeast)		Pore	Sandy	NM	1,229	NM	NM	NM	
		Pore	Sandy	NM	876	.3	NM	NM	
		Pore	Sandy	NM	692	.3	NM	NM	
	Site A	Pore	Sandy	NM	678	.3	NM	NM	
		Pore	Sandy	NM	2,053	.2	NM	NM	
		NM	Silt/clay	NM	NM	NM	NM	NM	
	Between sites	Pore	Silt/clay	NM	1,868	4.6	NM	NM	
	A and C	Pore	Silt/clay	NM	1,454	NM	NM	NM	
	Site C	Lake	Silt/clay	8.4	389	NM	24.3		

**Table 20.** Site characterization of Sullivan Lake microhabitat A (residential/septic influence) and water-quality values for pore-water samples collected July 8, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see fig. 2 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; mm, millimeters; °C, degrees Celsius; <, less than; NM, no field measurement; cm, centimeters]

			Location	(x, y) in m	
Constituent		(0, 1) (0, 3) (0, 5)	(5, 1) (5, 3) (5, 5)	(10, 1) (10, 3) (10, 5)	(15, 1) (15, 3) (15, 5)
Dissolved organic carbon	mg/L	3.0	2.6	2.1	2.1
		5.9	4.0	2.1	3.4
		3.2	7.4	3.6	3.7
Linear alkylbenzene sulfonates	mg/L	< .02	.29	.04	.16
		< .02	.92	.13	.13
		.38	.15	.04	.10
Alkylphenolethoxylates	mg/L	.14	< .02	< .02	< .02
		< .02	< .02	< .02	< .02
		< .02	< .02	< .02	< .02
Hydraulic head	mm	0	NM	.8	.1
		15	2.1	.1	.3
		3.1	1.4	.7	2.8
Temperature _{water/sediment}	°C	24.3	25.4	24.6	23.8
		25.1	25.6	25.0	23.0
		25.6	25.5	25.9	23.1
Temperature _{30 cm}	°C	22.9	21.7	21.2	16.5
		24.0	23.7	22.4	19.2
		23.4	23.6	23.4	20.5

**Table 21.** Water column water-quality measurements for Sullivan Lake microhabitat A (residential/septic influence) collected from 1:30 to 3:00 PM on July 21, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see fig. 2 for specific sampling-grid orientation; m, meters; total depth, distance from water surface to bed sediments; cm, centimeters; °C, degrees Celsius; µS/cm, microsiemens per centimeter; mg/L, milligrams per liter; std unit, standard unit]

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	42	10	29.1	390	8.9	8.5
		32	28.9	392	7.7	8.3
(3,5)	39	10	29.1	388	7.9	8.6
		29	28.7	390	7.9	8.5
(5, 5)	42	10	29.1	384	9.1	8.7
		32	30.0	385	8.8	8.6
(10, 5)	44	10	29.1	386	7.8	8.6
		34	28.9	386	7.8	8.5
(15, 5)	48	10	29.0	385	8.4	8.5
		38	27.4	414	5.8	7.9

**Table 22.** Site characterization of Sullivan Lake microhabitat B (residential/septic influence) and water-quality values for pore-water samples collected July 8, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see figure 2 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; mm, millimeters; °C, degrees Celsius; <, less than; NM, no field measurement; cm, centimeters]

			Location	(x, y) in m	
Constituent		NM (0, 3) (0, 5)	NM (5, 3) (5, 5)	(10, 1) (10, 3) (10, 5)	(15, 1) (15, 3) (15, 5)
Dissolved organic carbon	mg/L	NM	NM	7.6	9.4
		7.8	7.6	8.7	10.4
		10.3	10.0	11.4	10.1
Linear alkylbenzene sulfonates	mg/L	NM	NM	< .02	< .02
		.02	.04	< .02	< .02
		.12	.02	< .02	.03
Alkylphenolethoxylates	mg/L	NM	NM	< .02	< .02
		< .02	.10	< .02	.04
		.11	< .02	< .02	< .02
Hydraulic head	mm	NM	NM	-1	-3
		-2	0	-3	1
		3	-1	-1	0
Temperature water/sediment	°C	NM	NM	NM	NM
		NM	NM	NM	NM
		NM	NM	NM	NM
Temperature _{30 cm}	°C	NM	NM	NM	NM
		NM	NM	NM	NM
		NM	NM	NM	NM

**Table 23.** Water column water-quality measurements for Sullivan Lake microhabitat B (residential/septic influence) collected 10:30 AM to 2:00 PM on July 21, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see fig. 2 for specific sampling-grid orientation; m, meters; total depth, distance from water surface to bed sediments; cm, centimeters; °C, degrees Celsius; µS/cm, microsiemens per centimeter; mg/L, milligrams per liter; std unit, standard unit]

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	52	10	27.0	378	7.2	8.7
		26	26.8	378	8.0	8.7
		42	26.6	379	7.7	8.6
(3, 5)	54	10	27.1	377	7.8	8.7
		27	26.9	379	8.1	8.7
		44	26.8	378	8.7	8.7
(5, 5)	55	10	27.0	379	8.4	8.7
		28	26.8	378	8.7	8.8
		45	26.8	378	8.4	8.8
(10, 5)	60	10	27.2	378	8.8	8.7
		30	27.0	378	9.5	8.8
		50	26.9	378	8.9	8.7
(15, 5)	65	10	27.2	378	9.2	8.7
		22	27.0	378	9.3	8.8
		55	26.7	378	9.6	8.8

**Table 24.** Site characterization of Sullivan Lake microhabitat C (stormwater/boat-ramp influence) and water-quality values for pore-water samples collected July 8, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see fig. 2 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; °C, degrees Celsius; <, less than; NM, not measured; cm, centimeters]

		Location	(x, y) in m
Constituent	_	(0, 1) <b>NM</b>	NM (15, 5)
Dissolved organic carbon	mg/L	11.2	NM
		NM	7.3
Linear alkylbenzene sulfonates	mg/L	< .02	NM
		NM	.03
Alkylphenolethoxylates	mg/L	.11	NM
		NM	< .02
Temperature water/sediment	°C	23.7	NM
water sediment		NM	28.6
Temperature _{30 cm}	°C	19.4	NM
_ 50 cm		NM	17.7

**Table 25.** Water column water-quality measurements for Sullivan Lake microhabitat C (stormwater/boat ramp influence) collected 12:00 to 1:30 PM on July 21, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see fig. 2 for specific sampling-grid orientation; m, meters; total depth, distance from water surface to bed sediments; cm, centimeters; °C, degrees Celsius; µS/cm, microsiemens per centimeter; mg/L, milligrams per liter; std unit, standard unit]

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	82	10	27.1	384	9.2	8.4
		41	26.3	387	7.8	8.1
		72	26.2	392	5.2	7.8
(3, 5)	69	10	27.4	383	7.7	8.4
		35	26.8	385	7.8	8.3
		59	26.3	384	7.5	8.3
(5, 5)	68	10	27.4	381	8.4	8.4
		34	26.6	384	7.8	8.2
		58	26.2	389	6.9	7.7
(10, 5)	73	10	27.5	379	9.6	8.5
		37	26.5	384	9.1	8.1
		63	26.3	388	7.3	7.8
(15, 5)	71	10	26.9	379	10.1	8.5
		36	26.5	384	8.0	8.1
		61	26.1	394	5.9	7.5

**Table 26.** Site characterization of Sullivan Lake microhabitat D (agricultural influence) and water-quality values for pore-water samples collected July 9, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see fig. 2 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; mm, millimeters; °C, degrees Celsius; <, less than; NM, no measurement; * positive bias due to high inorganic carbon content; cm, centimeters]

		Location	n (x, y) in m
Constituent	_	(0, 1) (0, 3) (0, 5)	NM NM (5, 5)
Dissolved organic carbon	mg/L	31.8*	NM
		12.6*	NM
		32.3*	5.0
Linear alkylbenzene sulfonates	mg/L	.02	NM
		.11	NM
		.02	.11
Alkylphenolethoxylates	mg/L	< .02	NM
		< .02	NM
		< .02	< .02
Hydraulic head	mm	1.2	NM
		NM	NM
		NM	NM
Temperature _{water/sediment}	°C	22.9	NM
		23.8	NM
		21.7	19.9
Temperature _{30 cm}	°C	18.5	NM
***		18.8	NM
		17.4	16.7

**Table 27.** Water column water-quality measurements for Sullivan Lake microhabitat D (agricultural influence) collected 9:00 to 10:30 AM on July 21, 2010.

[x, distance parallel to shore; y, distance perpendicular to shore; see fig. 2 for specific sampling-grid orientation; m, meters; cm, centimeters; °C, degrees Celsius; mS/cm, microsiemens per centimeter; mg/L, milligrams per liter; std unit, standard unit; NM, no measurement]

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	70	10	25.1	388	4.1	7.4
		35	25.2	388	3.7	7.5
		50	25.1	389	3.6	7.5
(3,5)	72	10	25.5	880	4.3	7.6
		36	25.1	406	3.2	6.9
		52	25.0	443	2.9	6.7
(5, 5)	76	10	25.4	389	4.4	7.5
		38	25.2	450	2.9	6.7
		56	25.0	540	1.9	6.7
(10, 5)	71	10	25.8	385	5.1	7.9
		36	25.4	472	2.5	6.7
		51	NM	252	1.9	6.6
(15, 5)	78	10	26.1	384	5.6	7.9
		39	25.6	387	4.8	7.7
		58	25.2	451	3.0	6.8

Table 28. Summary of acidic organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on July 8, 2010.

[Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced;  $\mu$ g/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent; NM, no measurement]

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS Percent	Site B µg/L	Site B-Dup µg/L	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Blank µg/L	Blank µg/L	Blank µg/L
Ethylenediaminetetraacetic acid	0.2	0.2	63	0.2	0.2	0.2	0.2	68.6	0.6	0.6	< 0.05	< 0.05	< 0.05
Nitrilotriacetic acid	.1	.1	101	.1	.1	.1	.1	98.4	.1	.1	< .05	< .05	< .05
4-Nonylphenolmonoethoxycarboxylic acid	.6	.4	90	.4	.4	.4	.5	90.9	.4	.4	< .1	< .1	< .1
4-Nonylphenoldiethoxycarboxylic acid	.5	.1	95	< .1	< .1	< .1	< .1	90.0	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoltriethoxycarboxylic acid	< .1	< .1	80	< .1	< .1	< .1	< .1	78.1	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoltetraethoxycarboxylic acid	< .1	< .1	84	< .1	< .1	< .1	< .1	80.0	< .1	< .1	< .1	< .1	< .1
Surrogate Recovery (percent)													
4-normal-Nonylphenoldiethoxycarboxylic acid	69	66	95	68	72	68	87	85	79	75	98	NM	75

Table 29. Summary of acidic organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010.

[Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced; micropation per liter; <, less than; Dup, duplicate sample]

	Site	Site	Site	Site	Site
Compound	Α	A Dup	В	C	D
	μg/L	μg/L	μg/L	μg/L	μg/L
Ethylenediaminetetraacetic acid	0.3	0.3	0.2	0.2	0.3
Nitrilotriacetic acid	.1	.1	< .05	< .05	< .05
4-Nonylphenolmonoethoxycarboxylic acid	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoldiethoxycarboxylic acid	.2	.1	< .1	< .1	< .1
4-Nonylphenoltriethoxycarboxylic acid	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoltetraethoxycarboxylic acid	< .1	< .1	< .1	< .1	< .1
Surrogate Recovery (percent)					
4-normal-Nonylphenoldiethoxycarboxylic acid	64	46	55	70	52

Table 30. Summary of neutral organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn. on July 8, 2010.

[Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced;  $\mu$ g/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent; NM, not measured]

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS percent	Site B µg/L	Site B-Dup µg/L	Site B-MS percent	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Site D-MS percent
Acetylhexamethyltetrahydronaphthalene	< 0.01	< 0.01	111	< 0.01	< 0.01	114	< 0.01	< 0.01	131	< 0.01	< 0.01	124
Bisphenol A	.03	.02	67	.05	.01	68	< .01	< .01	85	< .01	< .01	84
2[3]- <i>tert</i> -Butyl-4-methylphenol	< .01	< .01	118	< .01	< .01	92	< .01	< .01	131	< .01	< .01	123
4- <i>tert</i> -butylphenol	< .01	< .01	111	< .01	< .01	116	< .01	< .01	129	< .01	< .01	129
Caffeine	.01	.02	31	< .01	< .01	31	< .01	< .01	30	< .01	< .01	41
2,6-Di-tert-butyl-1,4-benzoquinone	.03	.03	NM	.04	.03	NM	.04	.03	NM	.03	.03	NM
2,6-Di- <i>tert</i> -butyl-4-methylphenol	.04	.02	NM	.02	.01	NM	.03	.04	NM	.02	< .01	NM
2,6-Di- <i>tert</i> -butylphenol	< .01	< .01	136	< .01	< .01	98	< .01	< .01	146	< .01	< .01	143
1,2-Dichlorobenzene	< .01	< .01	99	< .01	< .01	98	< .01	< .01	88	< .01	< .01	111
1,3-Dichlorobenzene	< .01	< .01	98	< .01	< .01	96	< .01	< .01	88	< .01	< .01	110
1,4-Dichlorobenzene	< .01	< .01	98	< .01	< .01	96	< .01	< .01	87	< .01	< .01	110
<i>N,N</i> -Diethyl- <i>meta</i> -toluamide	.15	.14	107	.13	.13	118	.07	.08	117	.06	.06	125
4-Ethylphenol	< .01	< .01	73	< .01	< .01	69	.01	< .01	68	< .01	< .01	84
Hexahydrohexamethylcyclopentabenzopyran	< .01	< .01	107	< .01	< .01	107	< .01	< .01	124	< .01	< .01	118
5-Methyl-1H-Benzotriazole	< .01	< .01	<10	< .01	< .01	<10	< .01	< .01	<10	< .01	< .01	<10
4-Methylphenol	.02	.03	43	.03	.03	39	.03	.04	41	.03	.03	54
4-Nonylphenol	.09	.09	122	.05	.05	131	< .05	< .05	163	< .05	< .05	159
4-Nonylphenolmonoethoxylate	.28	.09	134	.09	.08	137	< .05	< .05	161	< .05	< .05	152
4-Nonylphenoldiethoxylate	< .05	< .05	110	< .05	< .05	117	< .05	< .05	137	< .05	< .05	129
4-normal-Octylphenol	< .01	< .01	12	< .01	< .01	12	< .01	< .01	15	< .01	< .01	14
4- <i>tert</i> -Octylphenol	.04	.02	137	.02	.01	138	.02	.01	162	< .01	< .01	157
4- <i>tert</i> -Octylphenolmonoethoxylate	.02	< .01	113	< .01	< .01	115	< .01	< .01	133	< .01	< .01	128
4- <i>tert</i> -Octylphenoldiethoxylate	< .01	< .01	109	< .01	< .01	108	< .01	< .01	122	< .01	< .01	119
4-tert-Octylphenoltriethoxylate	< .01	< .01	95	< .01	< .01	83	< .01	< .01	91	< .01	< .01	91
4-tert-Octylphenoltetraethoxylate	< .01	< .01	70	< .01	< .01	72	< .01	< .01	73	< .01	< .01	101
4- <i>tert</i> -Octylphenolpentaethoxylate	< .01	< .01	47	< .01	< .01	34	< .01	< .01	45	< .01	< .01	55
4- <i>tert</i> -Pentylphenol	< .01	< .01	122	< .01	< .01	128	< .01	< .01	147	< .01	< .01	142
4-Propylphenol	< .01	< .01	115	< .01	< .01	114	< .01	< .01	122	< .01	< .01	128
Triclosan	< .01	< .01	100	< .01	< .01	87	< .01	< .01	100	< .01	< .01	102
Surrogate Recovery (percent)												
d6-Bisphenol A	72	78		78	77		96	99		85	81	
d21-2,6-Di- <i>tert</i> -butyl-4-methylphenol	49	71		59	61		60	89		65	70	
4-normal-Nonylphenol	52	57		53	52		69	76		55	53	
4-normal-Nonylphenolmonoethoxylate	47	52		52	51		74	77		56	53	
4-normal-Nonylphenoldiethoxylate	29	39		41	40		59	63		45	40	

**Table 31.** Summary of neutral organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010. [Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat ramp influenced; microhabitat D, agriculture influenced; µg/L, microgram per liter; <, less than; Dup, duplicate sample]

Compound	Site A μg/L	Site A-Dup μg/L	Site B µg/L	Site B-Dup μg/L	Site C μg/L	Site D µg/L	Site D-Dup μg/L
Acetylhexamethyltetrahydronaphthalene	0.02	0.01	0.01	0.02	< 0.01	< 0.01	< 0.01
Bisphenol A	.01	.01	.15	.02	< .01	< .01	< .01
2[3]-tert-Butyl-4-methylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4- <i>tert</i> -Butylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
Caffeine	.02	.05	.04	.01	< .01	< .01	< .01
2,6-Di- <i>tert</i> -butyl-1,4-benzoquinone	.06	.09	.04	.06	.04	< .01	.04
2,6-Di- <i>tert</i> -butyl-4-methylphenol	.01	.01	< .01	.01	.01	.02	.02
2,6-Di- <i>tert</i> -butylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
,2-Dichlorobenzene	< .01	< .01	< .01	< .01	< .01	< .01	< .01
,3-Dichlorobenzene	< .01	< .01	< .01	< .01	< .01	< .01	< .01
,4-Dichlorobenzene	< .01	< .01	< .01	< .01	< .01	< .01	< .01
V,N-Diethyl- <i>meta</i> -toluamide	.09	.08	.09	.07	< .01	< .01	.02
-Ethylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
Hexahydrohexamethylcyclopentabenzopyran	.01	.02	< .01	.02	< .01	< .01	< .01
-Methyl-1H-benzotriazole	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-Methylphenol	< .01	< .01	.02	.01	< .01	< .01	< .01
-Nonylphenol	.06	.05	< .05	< .05	< .05	< .05	< .05
-Nonylphenolmonoethoxylate	.11	.08	< .05	< .05	< .05	< .05	< .05
-Nonylphenoldiethoxylate	< .05	< .05	< .05	< .05	< .05	< .05	< .05
-normal-Octylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-tert-Octylphenol	.09	.06	.05	.03	< .01	< .01	< .01
-tert-Octylphenolmonoethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-tert-Octylphenoldiethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-tert-Octylphenoltriethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-tert-Octylphenoltetraethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-tert-Octylphenolpentaethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-tert-Pentylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
-Propylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
riclosan	< .01	< .01	< .01	< .01	< .01	< .01	< .01
urrogate Recovery (percent)							
6-Bisphenol A	19	40	44	40	39	40	41
21-2,6-Di- <i>tert</i> -butyl-4-methylphenol	20	31	21	38	57	56	49
-normal-Nonylphenol	26	47	37	45	36	43	43
-normal-Nonylphenolmonoethoxylate	52	53	38	43	35	39	39
-normal-Nonylphenoldiethoxylate	44	53	47	53	43	49	49

**Table 32.** Summary of steroid and steroidal hormone compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on July 8, 2010. [Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced; μg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent; NQ, not quantifiable]

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS percent	Site B µg/L	Site B-Dup µg/L	Site B-MS percent	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Site D-MS percent
4-Androstene-3,17-dione	< 0.0001	< 0.0001	138	0.0009	0.0005	118	0.0005	0.0005	134	0.0014	0.0012	133
cis-Androsterone	< .0001	< .0001	150	< .0001	< .0001	139	< .0001	< .0001	130	< .0001	< .0001	150
Cholesterol	5.7	NQ	348	7.6	4.4	266	30	11	-664	23	NQ	327
Coprostanol	.029	.065	104	.042	.025	103	.053	.090	86	.20	.97	89
Diethylstilbestrol	< .0001	< .0001	97	< .0001	< .0001	66	< .0001	< .0001	97	< .0001	< .0001	97
Equilenin	< .0001	< .0001	105	< .0001	< .0001	86	< .0001	< .0001	101	< .0001	< .0001	107
Equilin	< .0001	< .0001	98	< .0001	< .0001	78	< .0001	< .0001	97	< .0001	< .0001	76
17α-Estradiol	< .0001	< .0001	117	< .0001	< .0001	132	< .0001	< .0001	112	< .0001	< .0001	119
17β-Estradiol	< .0001	< .0001	107	< .0001	< .0001	106	< .0001	< .0001	108	< .0001	< .0001	111
Estriol	< .0001	< .0001	115	< .0001	< .0001	73	< .0001	< .0001	62	< .0001	< .0001	235
Estrone	.0007	.0008	120	.0010	.0009	111	.0011	.0009	110	.0008	.0010	107
17α-Ethynylestradiol	< .0001	< .0001	110	< .0001	< .0001	100	< .0001	< .0001	103	< .0001	< .0001	104
Mestranol	< .0001	< .0001	107	< .0001	< .0001	99	< .0001	< .0001	106	< .0001	< .0001	106
Norethindrone	< .0001	< .0001	106	< .0001	< .0001	99	< .0001	< .0001	104	< .0001	< .0001	106
Progesterone	< .0001	< .0001	15	< .0001	< .0001	26	< .0001	< .0001	27	< .0001	< .0001	22
Testosterone	< .0001	< .0001	124	< .0001	< .0001	114	< .0001	< .0001	121	< .0001	< .0001	124
dihydro-Testosterone	< .0001	< .0001	130	< .0001	< .0001	109	< .0001	< .0001	123	< .0001	< .0001	113
epi-Testosterone	< .0001	< .0001	128	< .0001	< .0001	121	< .0001	< .0001	120	< .0001	< .0001	132
11-keto-Testosterone	< .0001	< .0001	81	< .0001	< .0001	69	< .0001	< .0001	95	< .0001	< .0001	91
Surrogate Recovery (perc	ent)											
d7-Cholesterol	47	<10		37	41		19	10		40	<10	
d12-Chrysene	105	106		183	115		115	119		105	104	
d8-Diethylstibestrol	59	<10		47	18		42	18		34	<10	
¹³ C ₆ -Estradiol	80	18		56	33		36	28		28	<10	
d2-epi-Estriol	42	18		46	49		39	19		16	18	
¹³ C ₆ -Estrone	96	30		75	97		71	65		97	31	
d4-Ethynylestradiol	82	26		62	71		47	49		70	25	
d3-Medroxyprogesterone	19	12		45	33		44	35		28	21	
d4-Mestranol	83	30		63	70		48	48		71	28	
d3-Nandrolone	63	24		54	61		54	45		30	17	

**Table 33.** Summary of steroid and steroidal hormone compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010. [Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced; µg/L, microgram per liter; <, less than]

Compound	Site A	Site B	Site C	Site D
Compound	μg/L	μg/L	μg/L	μg/L
4-Androstene-3,17-dione	< 0.0001	< 0.0001	< 0.0001	< 0.0001
cis-Androsterone	< .0001	< .0001	< .0001	< .0001
Cholesterol	3.3	3.4	2.8	3.2
Coprostanol	.042	.017	.017	.030
Diethylstilbestrol	< .0001	< .0001	< .0001	< .0001
Equilenin	< .0001	< .0001	< .0001	< .0001
Equilin	< .0001	< .0001	< .0001	< .0001
17α-Estradiol	< .0001	< .0001	< .0001	< .0001
17β-Estradiol	< .0001	< .0001	< .0001	< .0001
Estriol	< .0001	< .0001	< .0001	< .0001
Estrone	< .0001	< .0001	< .0001	.0006
17α-Ethynylestradiol	< .0001	< .0001	< .0001	< .0001
Mestranol	< .0001	< .0001	< .0001	< .0001
Norethindrone	< .0001	< .0001	< .0001	< .0001
Progesterone	< .0001	< .0001	< .0001	< .0001
Testosterone	< .0001	< .0001	< .0001	< .0001
dihydro-Testosterone	< .0001	< .0001	< .0001	< .0001
epi-Testosterone	< .0001	< .0001	< .0001	< .0001
11-keto-Testosterone	< .0001	< .0001	< .0001	< .0001
Surrogate Recovery (percent)				
d7-Cholesterol	39	46	53	15
d12-Chrysene	82	94	94	104
d8-Diethylstibestrol	80	69	83	52
¹³ C ₆ -Estradiol	63	65	58	50
d2-epi-Estriol	85	98	97	72
¹³ C ₆ -Estrone	118	116	106	81
d4-Ethynylestradiol	91	99	92	75
d3-Medroxyprogesterone	55	43	47	42
d4-Mestranol	103	101	94	74
d3-Nandrolone	121	96	98	74